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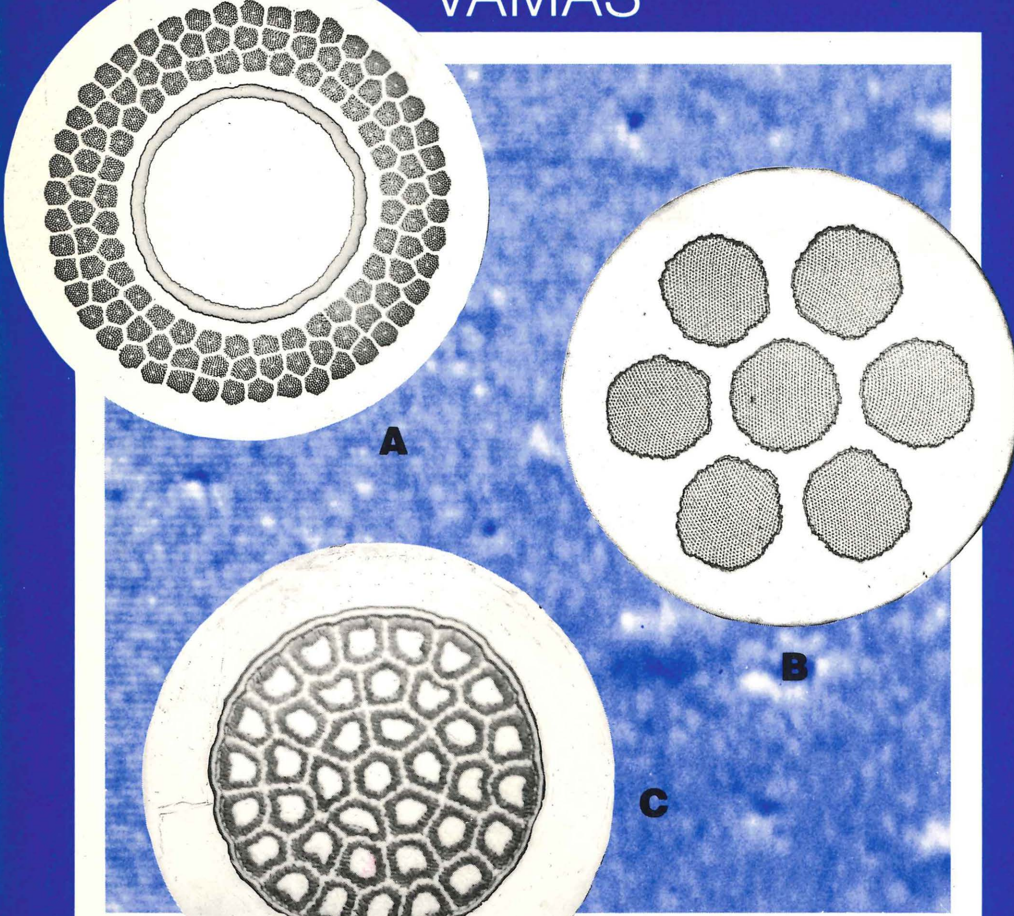

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Versailles Project on Advanced Materials and Standards
Canada • France • FR Germany • Italy • Japan • UK • USA • CEC

VAMAS

The Versailles Project of Advanced Materials and Standards (VAMAS) supports trade in high technology products through international collaborative projects aimed at providing the technical basis for drafting codes of practice and specifications for advanced materials. The scope of the collaboration embraces all agreed aspects of enabling science and technology - data bases, test methods, design methods, and materials technology - which are required as a precursor to the drafting of standards for advanced materials. VAMAS activity emphasizes collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardization action. Through this activity, VAMAS fosters the development of internationally acceptable standards for advanced materials by the various existing standards agencies.

VAMAS operates under a Memorandum of Understanding signed by senior representatives of government in the seven countries of the Economic Summits and of the Commission of the European Communities. The participating countries, Canada, France, and F. R. Germany, Italy, Japan, the U.K., the U.S., and the Commission of European Communities are each represented on its Steering Committee.

Cover: Cross sections of critical current measurement sample wires:
sample A - wire dia (0.8mm), number of filaments (~10,000)
sample B - wire dia (1.0mm), number of filaments (~5,000)
sample C - wire dia (0.7mm), number of filaments (~5,500)

Photograph courtesy of Prof. K. Tachikawa, Tokai University, Japan

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Activities in the Technical Working Area of Superconducting and Cryogenic Structural Materials

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Introduction

The application of superconductivity is expected to bring about a number of breakthroughs in a vast area of science and technology covering new energy systems, computer electronics, medical diagnosis, high-speed transportations, high-energy particle accelerators, etc. The recent discovery of high T_c oxide superconductors may provide a more positive prospect for the future development of the superconductivity technology.

The development of the superconductivity technology needs to be pursued under a long term concept, whereby international cooperation would be very effective and could make an essential contribution. To encourage such development, standard characterization methods must first be established as to relevant properties of superconducting and cryogenic materials necessary for superconducting equipment.

In response to the request by the VAMAS Steering Committee, an international technical working party (TWP) was organized, and TWP meeting has been convened four times since then, as listed in Table 1. The main guidelines that have been established through discussions at these meetings are as follows:

1. Round robin test programs on superconducting materials:
   - critical current (I_c) measurement on Nb_3Sn multifilamentary wires,
   - ac loss measurement on NbTi multifilamentary wires.
2. Round robin test programs on cryogenic structural materials:
   - tensile measurement at 4.2 K on SUS316LN and YUS170 steels,
   - fracture toughness measurement at 4.2 K on the same steels,
   - electrical strain gages at cryogenic temperatures.
3. Publication: a summary paper on the round robin tests by TWP may be followed by specialist papers.
4. New proposals: all new proposals should be submitted to the TWP office well in advance of the following TWP meeting.
5. Reorganization of TWP: formation of 2 subgroups -
   - Subgroup I for superconducting materials,
   - Subgroup II for cryogenic structural materials.

   The current TWP organization is schematically shown in Figure 1.
6. Annual TWP meeting; on the occasion of a relevant international conference.

Table 1. TWP Meeting Calendar

No	Site	Date	Number of participants
1	Kernforschunszentrum Karlsruhe, FRG	April 1986	19
2	National Bureau of Standards Boulder, USA	July 1987	18
3	Sunshine City Building Tokyo, Japan	May 1988	31
4	University of Southampton Southampton, UK	July 1988	20
5	University of California Los Angeles, USA	July 1989	

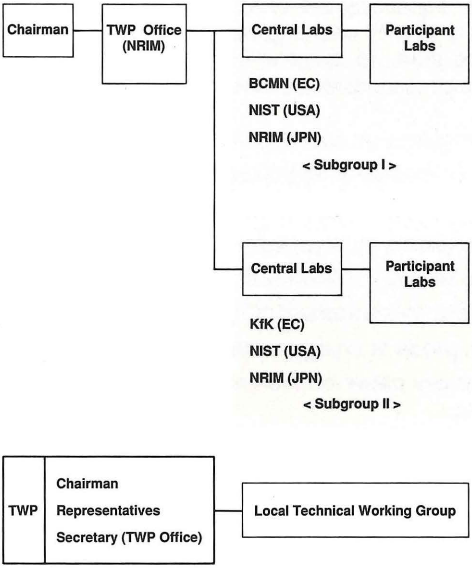

FIGURE 1. Current TWP organization.
Subgroup I for superconducting materials.
Subgroup II for cryogenic structural materials.

Round robin test on the critical current in Nb_3Sn wires

Critical current measurements on multifilamentary Nb_3Sn wires were successfully completed with 24 participant labs in Europe, USA and Japan listed in Table 2, and the results were intercompared to examine the effects of test variables, including specimen manufacturing processes, holder materials, measurement details, etc., on the scatter in measured I_c. One sample was supplied from each of three regions. The Bulletin cover shows cross sections of these 3 different samples, A, B, and C. To ensure a meaningful intercomparison, homogeneity in I_c properties along the sample wires was inspected, and found to be within ± ca. 1%, good enough for the intercomparison.

Figure 2 is an example of the test results that shows averaged I_c data and coefficient of data scatter on sample B as a function of magnetic field. The scatter in I_c reported spreads from 6 to 15%. This is rather larger than what may be estimated from the homogeneity test results, revealing that data scatter can be attributed to different measurement conditions and methods adopted by individual participants. Strain effect measurements on the test samples were also carried out, indicating that strains imposed on the samples may greatly change the I_c. This suggests that the sample mounting method including sample holder and bonding materials may have determining effects in the I_c measurement.

Round robin test on the ac loss in NbTi wires

Following the critical current measurement in Nb_3Sn wires, an intercomparison program for ac loss measurement on NbTi multifilamentary wires was started at 18 participant labs listed in Table 2. Taking into account a large variety of measurement techniques and the absence of a predominant practice, this program assumes that the measurement be carried out by means of existing techniques and on-site apparatus at each participant lab. Four NbTi sample wires have been supplied; JPN-A, JPN-B, European, and American samples. The JPN-A and American samples are for pulsed magnet and accelerator magnet use, respectively; and relatively large ac losses are expected. The JPN-B and European samples are for power frequency application and expected to have much smaller losses than the JPN-A and American samples.

This program is still underway, but preliminary results may be summarized as follows. The average hysteresis loss is 0.50 and 5.1 mJ/m for JPN-A and American samples, respectively, with corresponding standard deviations 0.06 and 0.6 mJ/m, respectively. These deviations are surprisingly small, in consideration of the current status of ac loss measurement. One European and two JPN-B solenoid samples are now being circulated among participant labs for measurement.

Table 2. List of Participant Labs in the Round Robin Tests on Superconducting Materials.

I_c Measurement	AC Loss Measurement
Europe	**Europe**
Atominst.Ost.Univ. (Austria)	Atominst.Ost.Univ. (Austria)
Inst.Expml.Phys.Ost.Univ. (Austria)	Alsthom D.E.A. (France)
S.C.K./C.E.N. (Belgium)	KfK (F.R.G.)
S.N.C.I., C.N.R.S. (France)	Siemens (F.R.G.)
KfK (F.R.G.)	C.I.S.E. (Italy)
Siemens (F.R.G.)	Univ. Twente (Netherlands)
Vakuumschmelze (F.R.G.)	Clarendon Lab. (UK)
E.N.E.A., Centro di Frascati (Italy)	
Univ. Nijmegen (Netherlands)	
Clarendon Lab (UK)	
Rutherford Appleton Lab (UK)	
USA	**USA**
Brookhaven National Lab	Battelle
Francis Bitter National Mag. Lab.	Brookhaven National Lab
Lawrence Livermore National Lab.	N.I.S.T.
N.I.S.T.	
Univ. Wisconsin	
Japan	**Japan**
Electrotechnical Lab.	Center Res.Inst.Elec. Power Industry
Furukawa Electric Co.	Electrotechnical Lab.
J.A.E.R.I.	J.A.E.R.I.
Hitachi	Kyushu Univ.
Kobe Steel.	Nihon Univ.
N.R.I.M.	N.R.I.M.
Osaka Univ.	Tohoku Univ.
Tohoku Univ.	Toshiba

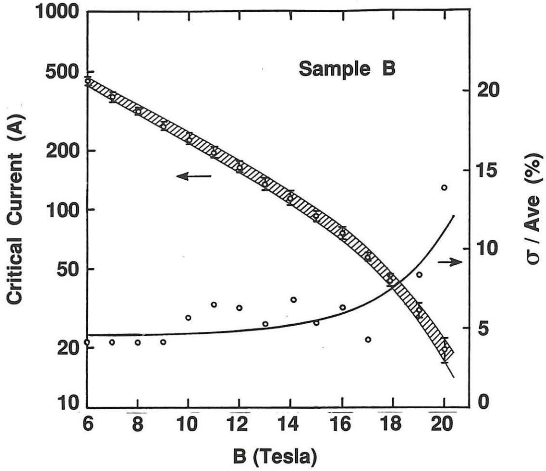

FIGURE 2. Averaged critical current vs magnetic field for sample B: υ (standard deviation) / averaged critical current is coefficient of scatter.

Round robin tests on cryogenic structural materials

Two austenitic steels, SUS316LN and YUS170 steels, were chosen as the round robin test materials. These steels are commercially available and have high yield strength and good fracture toughness favorable for cryogenic structure use. The participant labs in the tests are listed in Table 3. Tensile and fracture toughness test specimens were machined at the National Research Institute for Metals (NRIM) according to specifications designed by each participant. Each participant applied a different measurement method; there were no strict requirements on the measurement details, except that the strain rates were recommended to be low. Instead, detailed measurement conditions were reported along with the mechanical properties.

In general, tensile data reported are in a relatively good agreement with each other. It seems that the scatter in tensile strength measurement arises from load cell calibration error. Strain gages were found to give a larger scatter of data on the yield strength than did extensometers.

Table 3. List of Participant Labs in the Round Robin Tests on Cryogenic Structural Materials.

Tensile Measurement	Fracture Toughness Measurement
Europe	**Europe**
Techn.Univ. Wien (Austria)	KfK (F.R.G.)
KfK (F.R.G.)	
EMPA (Switzerland)	
Rutherford Appleton Lab. (UK)	
USA	**USA**
Lawrence Livermore National Lab.	N.I.S.T.
Mater.Res.Engr.Inc.	
N.I.S.T.	
Teledyne Engineering Service	
Japan	**Japan**
Hitachi	Hitachi
J.A.E.R.I.	Kobe Steel
Kawasaki Steel	N.R.I.M.
Kobe Steel	Tohoku Univ.
Nippon Kokan	Toshiba
Nippon Steel	Univ. Tokyo
N.R.I.M.	
Tohoku Univ.	
Toshiba	
Univ. Tokyo	

Figure 3 compares the scatter in yield strength data for the two methods. In the determination of elongation, we find that the influence of "serration", indicative of localized deformation, is important.

The fracture toughness measurement results are being analyzed. The scatter in fracture toughness data reported seems to be attributed to the measurement variables including the machine type, the strain rate, the deformation control-mode, etc.

In addition, a round robin test on the electrical strain gage calibration at cryogenic temperatures has been initiated with 13 participant labs.

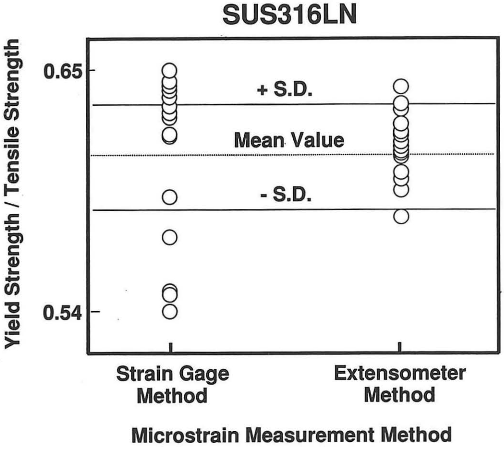

FIGURE 3. Comparison of scatter in yield strength normalized by tensile strength between strain gage and extensometer methods.

Remarks and Acknowledgements

A special VAMAS session has been established at the International Cryogenic Materials Conference (ICMC) scheduled at UCLA, for July 1989. More than 10 papers will be presented there. On this occasion, the 5th TWP meeting will also be held at UCLA.

New round robin test programs are being considered for different measurements and materials. These may involve strain effect measurement on superconducting materials, I_c measurement on large scale conductors, tensile, and fracture toughness measurement on Ti and Al alloys, etc.

It is very important in multilateral collaboration work like VAMAS to ensure smooth information flow from and to participants. In this respect, the TWP Office established at NRIM has occasionally been publishing a "TWP Office Report", to inform the participants of what is going on in the TWP and make them ready for such issues like new proposals to be discussed at the forthcoming meeting.

Finally, I would like to extend my sincere thanks to all the participants in the above round robin test programs and the Steering Committee members who have been supporting our activities.

RESULTS OF THE VAMAS CERAMICS HARDNESS TESTING ROUND-ROBIN

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Ceramics are becoming widely used for their properties such as hardness, wear resistance and good dimensional stability, and for this there is a need for some measures of performance. A round-robin has been designed and carried out to evaluate conventional metallurgical type hardness test methods using existing laboratory equipment but applied to very hard ceramic materials. The small size of indentations leads to possible large errors in hardness number, which can pose problems if hardness numbers are to be used as an agreed means of specifying a material. These conclusions were deduced from some earlier NPL work (1,2), from which this VAMAS study was initiated. The VAMAS round-robin had several aims, but was principally designed to determine typical distributions of data, and machine and/or operator biases when used under the extreme limits posed by ceramics.

Following participation by 20 organizations in the UK, France, F R Germany, CEC (JRC Petten), US and Japan, the results have been analysed by NPL, and the potential errors in test results quantified.

Round-robin design

Two high-alumina ceramics were prepared as a series of test discs with carefully polished surfaces, which were then divided into four zones. The two materials were chosen such that one, by virtue of its very small grain size (< 2 μm) and very low residual porosity, posed the minimum of observational or microstructure-limited difficulties; while the second was a conventional medium grain size two-phase material with about 3-4% of closed porosity. NPL made a series of HR45N, HV1.0, HV0.2 and HK0.2 indentations and recorded its data on the complete set of test discs (Figure 1). The participants were then asked to remeasure the HV1.0, HV0.2 and HK0.2 indentations with their own system, to make their own series of indentations and to report their results. Some of these second sets were then remeasured by NPL on return of the test discs, especially in cases where results deviated considerably from those originally made by NPL.

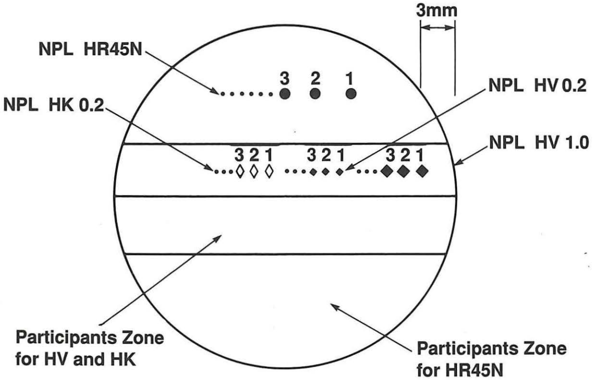


FIGURE 1. Layout of alumina test-pieces as received by the participants showing the positions of the NPL indentations and the zones for participants' own use.

The initial NPL indentations could be used as a reference set with which to compare the participants individually with the NPL observers, and to ensure that the set of discs behaved similarly. A comparison of NPL and participants' hardness test equipment can be made by comparing the two sets of participants' results. Analysis of the results has been performed to ascertain the biases and measurement errors likely in a typical population of users, few of whom had attempted in the past to make routine measurements of this kind on ceramics.

Results and discussion

The full results are reported in VAMAS Technical Report No. 3, and comprise an analysis of about 7000 individual measurements. A summary of the ranges and the averages of the data is given in Table 1. Most of the participants were able to make their own HV1.0 and HV0.2 indentations, but only about half were able to make HR45N and HK0.2 measurements. Nevertheless, all remeasured NPL's indentations.

TABLE 1 Summary of Results

	Material A HR45N	HV1.0	HV0.2	HK0.2	Material B HR45N	HV1.0	HV0.2	HK0.2
NPL indentations								
Mean indentation size, NPL, μm	-	31.0	13.4	38.8	-	38.9	15.9	43.6
Std. dev. of means, μm		±0.35	±0.24	±1.5		±0.60	±0.60	±2.1
Mean NPL hardness no.	88.2	1935	2058	1897	78.4	1228	1494	1494
Typical std. dev.	±0.3	±60	±70	±100	±0.5	±70	±100	±120
Range of participants' mean results, μm	-	29.6-32.6	12.1-14.2	35.2-42.9	-	36.9-41.3	15.0-17.7	41.2-48.2
Range of std. devs, ±		0.2-1.0	0.3-0.7	0.3-1.4		0.3-3.3	0.4-2.1	1.2-3.0
Mean participants' result, μm	-	31.4	13.4	38.8	-	39.4	16.1	44.2
Std. dev. of mean results		±0.9	±0.6	±2.3		±1.2	±0.6	±2.2
Mean participants' hardness no.	-	1885	2062	1917		1202	1462	1370
Range of std. devs. ±		20-129	59-203	30-172		21-253	96-355	84-191
Participants' indentations								
Range of mean indentation sizes, μm	-	29.8-34.1	11.6-14.3	36.0-40.4	-	37.9-42.8	11.8-17.9	42.3-47.1
Range of std. deviations, ±		0.1-2.4	0.2-0.8	0.2-1.6		0.5-5.5	0.5-3.8	1.3-6.7
Overall mean indentation size, μm	-	31.7	13.6	38.2	-	39.7	15.8	44.5
Std. dev. of mean		±1.1	±0.6	±1.2		±1.4	±1.3	±1.6
Overall mean hardness no.	89.0	1857	2038	1953	78.3	1190	1529	1470
Range of std. devs., ±	0.1-0.9	19-259	29-207	22-168	0.3-0.7	24-246	72-1242	82-372

Note: The differences in Rockwell hardness number shown above are exactly equivalent to the same value in μm, owing to the relationship HRN = 100 - δ where δ is the indentation in micrometres.

In considering the best method of analysing the results, the possible sources of error need to be borne in mind. With the optical reading methods (Vickers, Knoop), there are random errors due to real variations in true indenter penetration in the local microstructure, random errors of positioning microscope eyepiece crosswires, systematic errors due to variations between diamond indenter geometries, and also personal biases of judgement of the position of the indentation corners. Only the first and third of these sources are present in Rockwell tests, due to the mode of operation of the test machine; and since such indentations cannot be remeasured, simple averaging and determination of standard deviations are the most appropriate techniques. With the Vickers and Knoop tests it is desirable to establish whether individual participants, when compared with the experienced NPL operators, were able to follow size variations in indentations in the same way, or whether the random errors of measurement of either or both parties outweighed the real material variations. It was decided to use a covariance test on pairs of NPL and participants' results on individual indentations placed by NPL. Correlation coefficients obtained varied from near 1 to near -1, with the less-homogeneous material generally showing much better correlation than the homogeneous one. In other words, if there is a large variation in true indentation size as a consequence of microstructural inhomogeneity, then two independent observers are more likely to show the same trends of measurement than in the case of only small true variations, when random errors predominate. From these results it became clear that only when the standard deviation of individual indentation diagonal measurements exceeded about ±1 μm for Vickers indentations or about ±2 μm for Knoop indentations could two observers follow the same trend (Figure 2). Correlation coefficients less than +0.5 imply a high level of randomness in the measurements, while negative values have to be treated as freak cases of randomness.

Table 2 summarises the conclusions of the analysis for the two materials and the four tests. Material variability was low for the fine-grained alumina, but significantly higher for the two-phase alumina. Similarly, the difference between participant's and NPL's average measurements was significantly less for the fine-grained material. If it is assumed that the NPL observers were consistent in their measurement criteria throughout the project (their results were roughly in the centre of the spread from all participants (Table 1) suggesting their criteria were appropriate), then systematic participant biases could be as large as the typical standard deviations about their mean results. In contrast, differences between NPL and participant machines were small, as determined by the differences between participants' measurements of NPL's and their own indentations, except in the case of Knoop tests in which alignment of the diamond and orthogonality of loading direction to specimen surface are much more critical. The final figures in Table 2 are estimates

TABLE 2 Resolvability of Differences in Hardness

	Material A HR45N	HV1.0	HV0.2	HK0.2	Material B HR45N	HV1.0	HV0.2	HK0.2
Material variability as determined by NPL indentations, μm	±0.4	±0.5	±0.4	±1.5	±0.3	±1.0	±0.7	±2.0
Number of specimens employed	20	20	20	20	20	20	20	20
Single participant variability (typical standard deviations), μm	±0.4	±1.0	±0.4	±1.0	±0.5	±1.5	±1.0	±2.0
Number of indentations employed	5	5	10	10	5	5	10	10
Single participant bias, μm (typical NPL/participant difference)	±0.8	±1.0	±0.5	±0.8	±0.5	±1.5	±0.5	±1.5
Number of participants	6	20	22	21	6	20	22	21
Machine bias, μm, (difference between NPL's and participant's machines as measured by participants)	±0.8	±0.5	±0.3	±1.0	±0.5	±0.5	±0.3	±2.0
Difference in size needed to be resolvable by any two persons, μm	±0.9	±1.5	±0.7	±1.6	±0.7	±2.2	±1.2	±3.2
Number of indentations employed	5	5	10	10	5	5	10	10
Difference as % of mean size	±7.5	±4.6	±5.4	±4.3	±3.2	±5.7	±7.5	±7.1
Difference in hardness number	±0.9	±180	±220	±180	±0.7	±150	±230	±180

Note: The differences in Rockwell hardness number shown above are exactly equivalent to the same value in μm, owing to the relationship HRN = 100 - δ where δ is the indentation in micrometres.

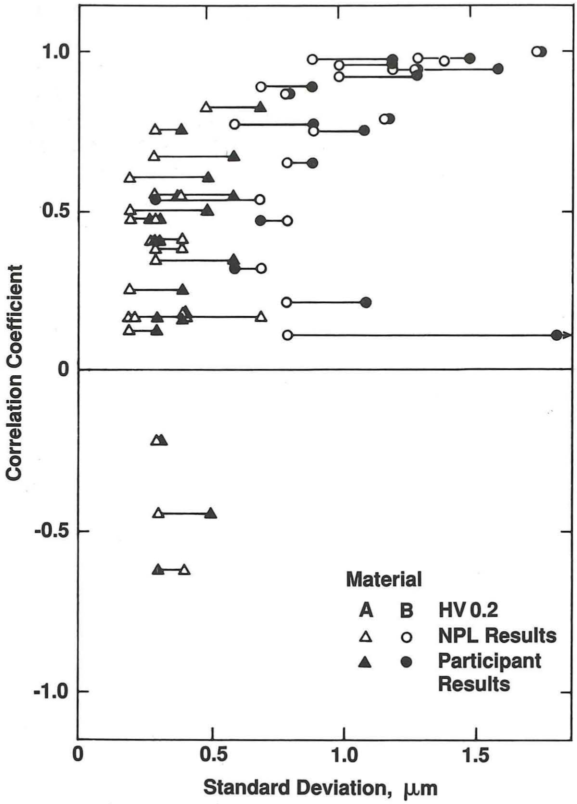
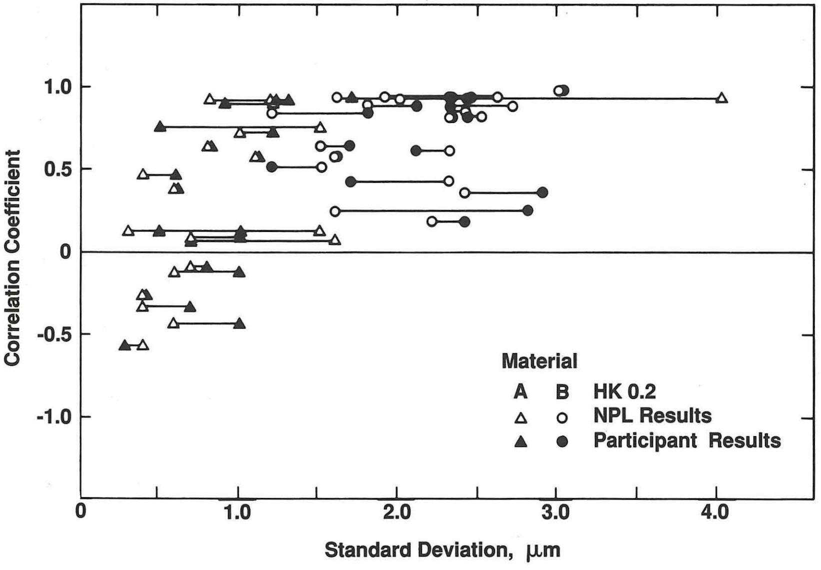


FIGURE 2. Relationship between correlation coefficients and standard deviations for measurements by NPL and by participants on individual NPL-placed indentations using (a) HV0.2 tests and (b) HK0.2 tests, showing that good correlation is obtained only if standard deviations recorded are larger than about 1 µm for HV0.2 and 2 µm for HK0.2.



of the true difference in mean indentation size needed before it can be declared resolvable by most participants on any test machine. These figures are a measure of the overall reliability of hardness data from any source. In this analysis it can be seen that possible combined errors in the visually measured tests are greater than 4% in indentation diagonal length, up to more than 7% in microhardness tests on the two-phase material. Such errors are doubled in calculation of the hardness number, giving the typical allowance that will have to be made in agreeing measurements or specifications. In the non-visual Rockwell measurements, the penetration of the indenter under the major load (δ) is recorded as the hardness number, i.e.

$$HR45N = 100 - \delta$$

This means that as the hardness of the material increases, penetration becomes less, and the hardness number becomes progressively more dependent on the exact shape of the indenter at its tip, and on the microstructure of the specimen under the indenter. For these reasons, potential errors increase as penetration becomes smaller. This conclusion is borne out by the results of the round robin which show that the typical differences between NPL tests and participants' tests are greater for the high-hardness material than for the two-phase material. This bias contributes a significant part of the total possible variation, given in the last line of the Table. Thus for Rockwell tests at less than 78 HR45N, the potential variation of results is the lowest of all the tests in this study, whereas at 88 HR45N, penetration is only one half of that at 78 HR45N, and the overall errors are similar to the maxima from all the other tests.

Conclusions

The VAMAS round-robin has enabled demonstration of the potential sources of error in the measurement of hardness of high-hardness ceramic materials. It has been shown that the combination of material variability and machine bias in HR45N tests results in possible uncertainties of about ±0.8 in the hardness scale, tending to be larger for hardness numbers approaching 90 than for those less than 80. In tests in which the operator has to make a visual judgement of the correct positions of the measuring cross-wires in a microscope, true indentation sizes in two materials must be different by at least 1 μm under good observational conditions, rising towards 2 μm or more for multiphase, inhomogeneous materials, especially when using the Knoop test. These factors combine in the calculation of a hardness number to give possible total errors (random observer and material errors plus participant bias) of typically ±10% with some participants showing even greater figures due to larger bias. These limitations need to be borne in mind if any attempt is made to standardise hardness testing for the purposes of classification or specification of ceramic materials. Further consideration should be given to the development of high-hardness reference materials, and to the examination of alternative methods of defining hardness of ceramics, e.g. automatic microhardness testers.

Acknowledgements

The author would like to express his thanks to the many participants who took part in the round robin, without whom it would not have been possible to assess all the factors involved.

1. Clinton, D. J., Morrell, R. "The Hardness of Alumina Ceramics," Proc. Brit. Ceram. Soc. **34** (1984), 113-27.

2. Clinton, D. J., Lay, L. A., Morrell, R. "The Microstructure Dependence of Hardness in High-Purity Alumina Ceramics." Proc. Brit. Cer. Soc. **37** (1986), 217-27.

The VAMAS International Workshop on Standards for Material Databanks

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Materials databases are growing in importance as more and more companies computerize their technical activities. From the start, groups building and using materials databases have recognized that good standards would significantly reduce their work and would make sure that data from different databases could be compared and combined as desired. National and regional standards organizations have begun efforts to develop the necessary standards. As the work proceeds, it is apparent that actions must be taken to coordinate and harmonize national standards whenever possible.

To this end, the VAMAS Technical Working Party 10 on Material Databanks has been working to identify the needs for standards, to establish priorities and to foster action. In 1987, the VAMAS Report on "Factual Materials Databanks -The Need for Standards"[1] outlined four major areas for standardization:

- Basic considerations for handling data
- Material data generation and reporting
- Materials databases
- Access to materials data

Numerous specific recommendations were made to different organizations. However, the report did not set any specific priorities; nor were any action plans proposed.

To start work on these recommendations, VAMAS brought together 45 experts from 13 countries to an international workshop in November 1988 at Petten, Netherlands. The goal of the workshop was to define the next standards actions to ensure that the materials databases and information networks currently being built would effectively serve their users.

Action steps were identified for five standardization areas where the needs for cooperation are most acute.

1. Materials identification
2. Terminology harmonization
3. Data exchange
4. Data reporting formats
5. Models for data evaluation and analysis

[1] Factual Materials Databanks - The Need for Standards, H. Kröckel, K. Reynard and J. Rumble, Eds. (Available from Standard Reference Data, NIST, A323 Physics Building, Gaithersburg, MD 20899).

Materials Identification

Because materials databases primarily contain properties of a **material**, how that material is described in a computer database is very important. The identification scheme used must meet two goals. An individual material must be uniquely identified; and the scheme must allow the determination of the equivalency of two materials so that data sets can be combined.

Identification schemes must not be confused with materials numbering systems such as the Unified Numbering System for metals (UNS). The numbering systems represent just a small part of the overall scheme for materials identification.

The VAMAS Workshop established that for a materials identification system to be successful, three criteria must be met: 1) it must be international, 2) it must be possible to translate to and from existing systems, 3) the procedures to add new materials and to administer the system must be easily implemented.

To establish a coherent identification scheme for databases on engineering materials (polymers, ceramics, composites, and metals), a coordinated international effort is needed. The first step is to use schemes already proposed as the basis for discussion by national, regional and international standards groups. To that end, a series of workshops are to be organized to discuss proposed schemes to refine them and ensure that all points of view are considered. This activity has already started. ASTM Committee E49 held the first workshop, focused on numbering systems, in February 1989 to coordinate efforts within the United States.

VAMAS has two related projects underway. One is identifying existing designation systems, under the leadership of Keith Reynard (UK) with support from the Department of Trade and Industry (UK). A second effort (VAMAS TWP 14) led by Sam Schneider of NIST (US) is working on a ceramics identification scheme.

Terminology Harmonization One feature of computerized information in databases is the need for complete definition of all terms, including synonyms. On the printed page, the eye and mind can make rapid substitution of one term for another—elastic modulus for Young's modulus—but in computerized systems, software must be programmed to do that.

There are two approaches: 1) to force the use of a controlled vocabulary, and 2) to provide a thesaurus that contains all terms and their synonyms. Neither approach is satisfactory by itself, and a combination is needed. The individual concepts behind the terms need to be standardized to the fullest extent possible and existing definitions harmonized. Equivalency tables can then be set up for **important** concepts and terms.

The Workshop recognized that harmonization can proceed only by building upon the existing activities, most importantly those of ISO, ASTM, CEC/CEN and the Pacific Rim Countries. Three specific recommendations were made.

1. ISO standard definitions should be viewed as the first choice.
2. Experts on materials databases terminology questions from ASTM, CEC/CEN and the Pacific Rim Standards organizations should meet with ISO to determine a strategy for broadening and consolidating the ISO definitions. The work would include:

- Making a compilation of ISO terms and definitions relevant to materials databases
- Making a compilation of all existing terminology systems relevant to materials databases
- Defining a mechanism for identifying missing terms and preparing definitions for endorsement by ISO

3. A uniform format for a terminology database should be developed based upon what has been done by ISO Eurodicatom and ASTM Committee E49.

Data Exchange

The exchange of materials data in computerized form is very important because many of the uses themselves are computerized, and materials data are often just a small part.

Materials data exchange plays a role in two large information exchange activities that have been standardized for commercial transactions and for product information exchange. ANSI Committee ASC X12 and ISO Technical Committee 184 are actively developing standards for commercial transactions, especially those involving purchasing and shipping. The Product Definition Exchange Specification (PDES) being developed by ISO Technical Committee 184 covers standards for design and manufacturing information such as geometrical or finite element analysis. Both efforts include materials data in their overall scheme, but the two systems are not sufficiently compatible.

Materials database builders, distributors, and users have their own needs for data exchange, concentrated in two areas: data entry and database exchange. A format for data entry will allow data files prepared to this standard to be used by different database builders for input into their databases. Database exchange will enable materials database networks to integrate easily different databases as well as ensure that data from different databases can legitimately be combined and compared.

The workshop concluded that coordinated international action is needed to address both of these concerns. The proposed action is for a materials data interchange format to be prepared for test. This is to be done on an international scale; a meeting in late 1989 should discuss the results of that testing. This effort will be headed by Philip Sargent of Cambridge University under the auspices of BSI and ASTM and will include representatives from several groups.

Data Reporting Formats

Materials databases generally contain property data for materials. Standards for description of the materials have already been discussed. Standards for reporting property data are equally important. The strategy adopted consists of two parts: one to develop generic guidelines for reporting of property data and a second to write detailed formats for individual test methods.

The Workshop recommended that the generic guidelines be reviewed, extended, and adopted on an international basis. This work could proceed directly through submission to ISO, or with parallel development by national and regional standards groups. Following this, differences could be resolved on the ISO level. The path chosen will depend on how vigorously national standards bodies propose to move ahead.

Modeling and Methods for Data Analysis and Evaluation

Materials data are rarely intrinsic properties, but rather arise from measurements made according to a prescribed test procedure. Property data are generated from the analysis and evaluation of raw test data using models and methods developed over the years. Correct analysis requires the specification of many different data items, a requirement that is sometimes overlooked.

The VAMAS workshop felt that high-quality materials databases were possible only if more careful attention were paid to analysis and evaluation. It was proposed that VAMAS undertake the development of an inventory of the models and methods used for data analysis and evaluation. The first step would be to identify the data needed for each model and method. A longer-term goal would be to harmonize these with respect to description of required input data.

These tasks need to be done from the perspective of the end user. Information would include materials group or class, properties to be evaluated, models for the property, parameters to be used as input in the model, and limitations of the model. These would give standards bodies the basis for further work on standardizing the models.

What will happen next?

The specific recommendations made here have been sent to specific groups and their leaders. The proposed work is important and has high impact. Most can be done with fairly limited resources. If the goals of harmonized materials databases are to be realized, then this work is necessary.

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• TECHNICAL WORKING AREAS •

Technical Working Area 1

WEAR TEST METHODS

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A second round robin comparison on the reproducibility and comparability of friction and wear tests with participation from 29 institutions from all VAMAS member countries and also Denmark and Finland has been performed with combinations of α -Al_2O_3 ceramic, Si_3N_4 ceramic, and AISI 52100 steel under conditions of dry sliding. For like pairings of these materials, significant materials-related differences of the friction (f) and the wear (W) were found in the following ranking order:
f (Al_2O_3/Al_2O_3) < f (steel/steel) < f (Si_3N_4/Si_3N_4) and
W(Al_2O_3/Al_2O_3) < W(Si_3N_4/Si_3N_4) < W(steel/steel).
Where there are unlike materials pairings, depending on the ratio of the contact area to the total wear track, the stationary and the moving components of the test system respond differently to the interfacial tribological action. Various interfacial tribological processes, e.g., microfracture, ploughing, materials transfer, tribooxydation, and corresponding appearances of the wear surfaces were identified by means of scanning electron microscopy (SEM) and energy dispersive analysis of X-rays (EDAX). If, for a given unlike material pairing, the materials of the stationary and the moving partners are exchanged, a significant variation in the system wear as well as in the wear of the two components may result.

The great importance of the proper definition and control of component and system parameters of the materials' bulk and surface conditions, the contact geometry, and the operating environmental conditions for the reproducibilty of the tribological tests were clearly demonstrated. For an unambiguous report of wear data, values related to the wear system and to the two individual materials components of the system must be distinguished. From the results of these multilaboratory studies, conclusions for the performance of reproducible tribotests can be drawn. The results of the VAMAS exercise were presented as an invited plenary lecture at the 7th International Conference on Wear of Materials, Denver, April 10 to 14, 1989.

Technical Working Area 2

SURFACE CHEMICAL ANALYSIS

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A number of significant developments have occurred within this Technical Working Party activity which maintain its initial momentum. Referring to Figure 1 of Bulletin No. 9 of January 1989 we see that two projects had been completed, numbers 1 and 10. As suggested in Bulletin No. 9, as projects become complete they will spawn new ones. The material in Project 1 is nearly exhausted and so new material is being established on a new instrument which has led to some marked improvements in depth resolution data. The completion of Project 10 has provided us with a file format to transfer data; but, in the general transfer of material, there are further hurdles concerned with standardizing our procedures of distribution to ease communication.

Four projects: 2, 9, 16 and 17 have data being analyzed. These analyses are nearing completion, and in all cases the results indicate very positive outcomes. In Project 2 the results of the coordinated intercomparison show that spectrometer energy scales may be calibrated, using a copper reference standard, to levels close to the instrument repeatability. The resultant scatter shows that a factor of six improvement may be obtained in most instruments and a factor of twelve in high resolution instruments, since the 1982 ASTM survey. During the next period this report will be circulated for comment and NIST and NPL will prepare a joint recommendation for the calibration of energy scales. In Project 9 even greater improvements appear to be possible, and the procedure for intensity calibration appears to be valid for most spectrometers at the 2% standard deviation level. In Project 16, a very extensive analysis of the data for measuring peak positions and for comparing angle resolved XPS and ion sputter depth profiles to estimate overlayer thicknesses shows an excellent level of consistency. Finally, Project 17 has established a high level of correlated AES data for Au/Cu alloys such that much is now known about their interpretation. As this project too draws to a close a new project, based on these alloys, will be spawned using XPS.

One new project, originally listed as Project E has now been incorporated into the main program. The remainder of the projects are developing strongly in their research and coordination stages. In some of these, particularly those involving the development and assessment of computer algorithms, such as Projects 13 and 18, computer programs are starting to be distributed to interested participants for assessment, development, and validation.

New inter-project coordinations are being developed which further strengthen the individual project's contribution. A general call for involvement in the SNMS project, Project 15, has gone out to relevant researchers; we note also that SNMS will be used to characterize the reference oxides in Project 8. Thus, in reverse, the characterized oxides may be used in homogenizing SNMS data in Project 15. Again, in Project 14 the reference alloys of Project 17 provide some interesting and crucial tests of the validity of the general algorithms developed.

Project Status

Details of the projects can be found in more detailed reports, which are distributed among participants as follows:

Canada	= 12, 13
France	= 7, 11, 16
FR Germany	= 5, 8
Italy	= 15
Japan	= 17, 19, 20
UK	= 1, 2, 9, 10, 21
USA	= 2, 3, 18
CEC	= 14

Technical Working Area 3

CERAMICS

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Reports on recent intercomparisons in Fracture and Hardness are being prepared.

Technical Working Area 4

POLYMER BLENDS

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This report is being prepared a couple of weeks before the 5th Annual Meeting of VAMAS Technical Working Party of Polymer Blends (TWP-PB) to be held this year in Kyoto, Japan. As always, the meeting will provide an occasion for reviewing the technical progress as well as for discussions on development of the program. The complete information will be given in Bulletin No. 11.

Phase I of the program is completed and the remaining four final reports are in the last stage of preparation. Phase II was formally initiated last November when two tons of Orgalloy, the crystalline/crystalline polyamide-6/polypropylene (PA/PP) commercial blends, were supplied and distributed to all TWP-PB members by ATOCHEM. The material is available in granular form and as 2 mm thick extruded sheets; the optimal geometry for the mechanical testing is being evaluated in the UK. Preliminary measurements have been carried out on: flow behavior at 225 and 250°C, morphology (before and after shearing), thermal analysis, dynamic tests in solid state, etc.

In addition, a sample of amorphous/amorphous polymer blend, the high impact polystyrene (HIPS) form Dow Chemical, is being evaluated for its impact performance. The first morphological scans indicate that the rubber particle diameter is about 0.3 µm, which should ensure the desired brittle behavior.

The third commercial blend also evaluated for suitability in Phase II tests is "natural" Ultrablend KR 4080 from BASF. The mixture of compatibilized, amorphous polycarbonate and crystalline polybutyleneterephthalate (PC/PBT), has particle size not exceeding 0.3 µm.

Encouraging news is also coming from France and the UK where the National TWP-PB are particularly active. The organization in France held its meeting on December 7, 1988 to plan for Phase II activities. In the UK, where most of the preliminary tests on suitability of HIPS and PC-PBT blends are carried out, the mechanical testing on PA/PP blends are at an advanced stage. Last fall, a formal link between the European Group of Fracture and TWP-PB was established through the UK TWP-PB.

Technical Working Area 5

POLYMER COMPOSITES

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The aim of the VAMAS composites program is to evaluate the mechanical properties of composite materials by delamination, fatigue, and creep testing. Three round robins are underway.

Delamination Testing

In cooperation with ASTM D30.02.02 the VAMAS working group is developing a specimen specification and a testing method to determine toughness criteria and fatigue delamination growth in tension (mode I) and in shear (mode II).

Tests have been carried out on unidirectional glass fiber epoxy composites and equilibrium woven composites from Vetrotex and T 300, T 800 carbon fiber epoxy composites from Toray. In order to promote standardization of the monotonic delamination test in mode I, first results were published since 1988. Fatigue delamination tests started early 1988 and will last through 1989.

Fatigue Testing

A program on fatigue testing is being conducted in order to establish reliable specification and testing methods for use in prediction of the fatigue limit of glass and carbon fiber composites. An important topic is to compare pure tension loading and bending loading tests. Specimens are supplied by Permali, Toray, and Vetrotex. The leader of this working group is Dr. Sims from NPL (U.K.).

Creep Testing

A new round robin was born the last year in order to prepare the standardization of creep testing. The program involves creep tests in tension and in flexure with two types of specimens made in carbon fiber epoxy composite supplied by Toray. The leader of the working group is Dr. R. Kemmochi from IPRI (Japan).

Technical Working Area 6

SUPERCONDUCTING AND CRYOGENIC STRUCTURAL MATERIALS

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Recently, ultra-fine filamentary Nb-Ti wires useful for AC applications are of growing importance. A round robin test of AC loss measurement in these wires is proceeding among 18 participant labs. Electro-magnetic measurements are performed on Nb-Ti wires with relatively large AC loss, whilst calorimetric measurements are performed on those with very small AC loss. The intermediate result of the round robin test indicates that the coefficient of standard deviation in AC loss is about 12%, which is rather small in view of the variety in measurement apparatus.

The results of round robin test on critical current measurement in Nb_3Sn wires among 24 participant labs (9 countries) have been summarized, and papers will be published shortly.

In the area of cryogenic structural materials, the round robin test on tensile testing at 4.2 K using SUS 316LN and YUS 170 steels was completed, and a summary report has been published. Following this, the round robin test on fracture toughness at 4.2 K using the same steels has been recently completed. A round robin test on strain gauge calibration at cryogenic temperatures proposed by Dr. Pavese of IMGC has been newly started with 13 participant labs.

A special session will be set for the VAMAS activities of this area at the International Cryogenic Materials Conference to be held in Los Angeles in the end of July. The next TWP meeting will be arranged also at this occasion.

Technical Working Area 8

HOT SALT CORROSION RESISTANCE

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Preparations for an international intercomparison to probe the validity of the draft document giving guidelines on hot-salt corrosion testing procedures are now well advanced. Batches of test pins have been cast in René 80 and IN738 according to the requirements of the participants and coating of some of the pins is now in progress. When all the materials are available, samples will be dispatched to each participant so that the test programme can proceed.

In the context of the general interest in test methods for hot-salt corrosion of gas turbine materials a special issue of the journal "High Temperature Technology" is being planned, this will feature, among other items, an account of the VAMAS activities in this area.

Technical Working Area 9

WELD CHARACTERISTICS

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In this programme the first phase of the work involves an intercomparison of weld profiles obtained in TIG-welded samples of the austenitic steels 304 and 316. Initial results were discussed at a coordination meeting held at NPL, UK in December 1988; and the importance of surface-tension driven flow in controlling weld penetration was confirmed. However certain aspects of the arc-characteristics also influence the dynamics of the weld pool, particularly the electromagnetic and aerodynamic forces. A mathematical modeling procedure is being developed in Japan to aid in the investigation of the relative influence of the various parameters that can effect the weld profile. Experimental work to validate the model is in progress. Results presented from the USA were consistent with those obtained in the UK and Japan in confirming that better weld penetration occurred in the higher sulphur steels.

Participation from the Joint Research Centre in Petten, The Netherlands, was welcomed. In concentrating on laser welding, this work will avoid the complications associated with the effect of electromagnetic forces. A representative from the International Institute of Welding (IIW) was present at the meeting and confirmed the interest of that organization in the Project. It was agreed that a report of the intercomparison should be prepared for publication in the Journal of the IIW.

Technical Working Area 10

MATERIALS DATABANKS

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This group has recently completed one prestandardization project and has two other projects underway. The VAMAS International Workshop on Standards for Materials Databanks held on November 15-17, 1988 at the Joint Research Centre in Petten, the Netherlands was attended by 45 persons from 13 countries. A report of this meeting is given elsewhere in this Bulletin.

Two other projects on Materials Designation Systems and Data Evaluation are underway. An inventory of materials designation systems is being compiled under the leadership of Dr. K. Reynard of the U.K. This project has been delayed because of slowness of funding. Money is now available and rapid progress is expected. The round-robin comparison of data evaluation methods for creep and fatigue data for steel alloys is nearing completion under the leadership of S. Nishijima of Japan.

Technical Working Area 11

CREEP CRACK GROWTH

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The first phase of this programme, concerned with ferritic steels, has now been completed. The overall conclusion is that an encouraging level of agreement can be obtained in creep crack growth measurements provided consistent experimental and analytical techniques are used. The work has provided that basis for agreement on a standard test procedure; and initial consideration has been given to identifying the most suitable route for producing such a document. The completion of the first phase of the programme has been marked by the publication of a state-of-the-art report which is now available in final form.

As a result of the planning meeting, held at Houston, Texas on the occasion of the recent ICF7 Conference, the main themes to be considered in the next phase of the work have been agreed. These are: **1.** Extension of the approach adopted in phase one to a wider range of materials; **2.** Analysis and modeling of the early stages of crack growth; and **3.** The unambiguous determination of the deformation mode in typical specimens. These activities will be carried out collaboratively by the relevant groups in Japan, Europe and the USA. The next coordination meeting at which progress will be discussed will probably be held in Europe early in 1990.

Technical Working Area 12

EFFICIENT TEST PROCEDURES FOR POLYMER PROPERTIES

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The initial work program which was agreed at the May 1988 meeting of TWA 12, and which was reported in Bulletin No. 9, is now well under way. The five participating nations (UK, US, France, FR Germany, and Japan) have conducted assessments of their countries' uses of accelerated durability testing, identifying preferred tests, their principal features, the reasons for their use, the needs for improvements and the potential for harmonization. These separate national studies are being compared and combined into a VAMAS report in which the need for further pre-standards work by VAMAS will be identified. Studies on the status of analytical methods for predicting long term performance from data from short term accelerated tests are being progressed, and all aspects of the work will be discussed at the next TWA 12 meeting in September 1989.

Technical Working Area 13

LOW CYCLE FATIGUE

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The results of this study, which is aimed at examining the effects of testing variables, are currently being collated and analyzed. A summary of the main conclusions will appear in a focus review in the next issue of the Bulletin after which a detailed report will be available.

Technical Working Area 14

THE TECHNICAL BASIS FOR A UNIFIED CLASSIFICATION SYSTEM FOR ADVANCED CERAMICS

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This activity, initiated in September 1988 by approval of the VAMAS Steering Committee, has three prime objectives: **1.** to identify and assess the issues inherent in developing a classification system for advanced ceramics; **2.** to establish a building-block structure for international use; and **3.** to develop mechanisms and institutional links for system implementation.

The Technical Working Group now has been fully constituted with each of the VAMAS member nations having representation. Progress has been made both in project organization and preliminary data gathering. The Working Group held its first meeting on April 6-7, 1989 at Bundesanstalt für Materialforschung und -prüfung (BAM) in West Berlin to map out strategy and set plans for project implementation. The agenda included a review of classification systems for conventional materials, the development of a survey questionnaire on advanced ceramics classification needs, scope and basis, and the holding of a general interest classification workshop. The following summarizes major discussion points and conclusions reached:

1. A tentative "working definition" of advanced ceramics was formulated to set the range of products to be covered by the classification system. A broad scope was adopted and includes all major functional types, i.e. structural, electronic, magnetic, etc.

2. Preliminary review of conventional materials classification schemes indicated that they only marginally provide classification models for advanced ceramics. Current systems (e.g. metals) are based on a compositional framework, whereas a functional use (application) might be more appropriate for advanced ceramics.

3. A review was made of a classification survey questionnaire recently distributed to members of the United States Advanced Ceramics Association (USACA). It was decided that it was suitable for circulation in Europe, Canada and Japan. Each working group member will handle distribution to key industrial companies within their country. Responses will be complete by August 30, 1989.

4. It was agreed to hold a classification workshop, tentatively scheduled for June 21-22, 1990 at either Petten Research Centre, Netherlands, or Ispra Research Centre, Italy.

5. The next meeting of the Working Group will be held December 11-12, 1989 at the National Physical Laboratory, Teddington, England. The Working Group encourages participation by other interested persons.

• VAMAS CALENDAR •

Surface Chemical Analysis Technical Working Party, in conjunction with Eleventh International Vacuum Congress and Seventh International Conference on Solid Surfaces, Köln	September 25-29, 1989
Topical Conference on Quantitative Surface Analysis at Salem, Massachusetts, prior to 3th AVS meeting 24-27 October 1989, Boston	October 20-21, 1989
Surface Chemical Analysis Technical Working Party, in conjunction with ECASIA 89, Juan Les Pins	October 24-27, 1989
Surface Chemical Analysis Technical Working Party, in conjunction with AVS Meeting, Boston	November 3-7, 1989
The Technical Basis for a Unified Classification System for Advanced Ceramics, Teddington	December 11-12, 1989
Ceramics Classification Workshop, Petten	June 21-22, 1990
Quantitative Surface Analysis, QSA6, Royal National Hotel, London	November 12-16, 1990

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An HR45N indentation in a fine-grained high-alumina ceramic showing radial cracking outside and fragmentation inside the indented area. See article by Roger Morrell.