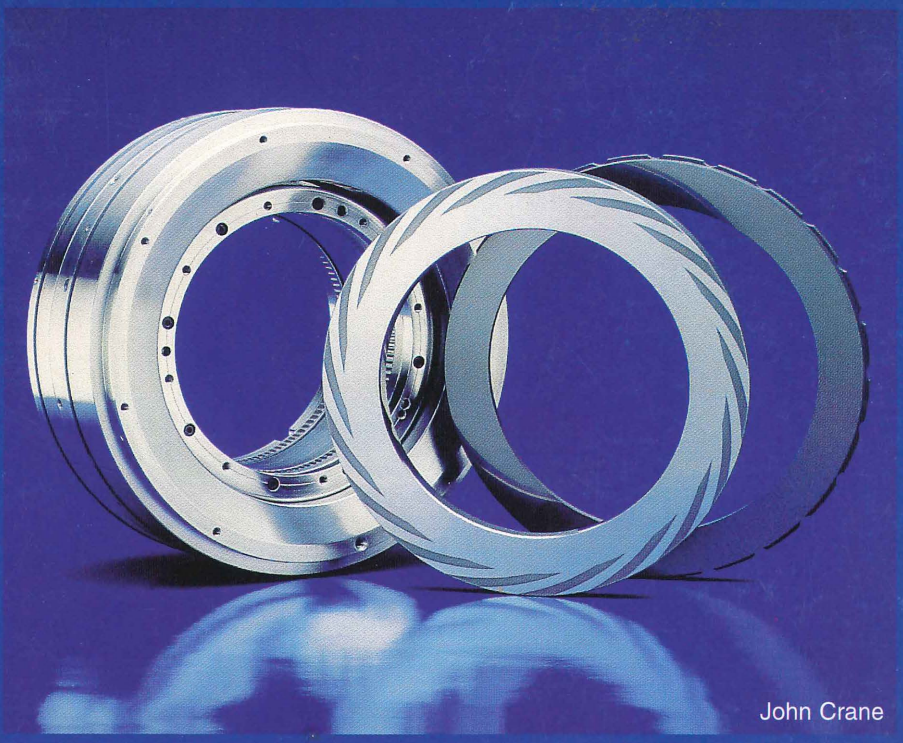
VAMAS



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April 1999

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

VAMAS

The Versailles Project on 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 - databases, test methods, design methods, and materials technology - which are required as a precursor to the drafting of standards for advanced materials. VAMAS activity emphasises collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardisation action. Through this activity, VAMAS fosters the development of internationally acceptable standards for advanced materials by the various existing standards agencies.

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Cover: John Crane dry running non contacting gas seal. The groove pattern on the rotating hardmetal seal pumps gas inwards towards the non-grooved portion, thus preventing face to face contact and wear during operation. Hardmetal was chosen because of its high strength, high toughness and high thermal conductivity.

Photograph courtesey of John Crane International

VAMAS

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• Foreword •

The Chairmanship of VAMAS and the Secretariat will be passing to the USA during the next Steering Committee meeting at the end of May in Canada. The new Chairman will be Steve Freiman who has been a national representative on the Steering Committee for the last four years and the Secretarial responsibilities will be in the experienced hands of Jim Early who will be taking on this role for the second time.

My involvement with VAMAS has spanned over 10 years with two most rewarding periods as Chairman. Now that the MoU for VAMAS has been renewed with no specific time limit, it is vital for us to ensure that VAMAS continues to operate as a dynamic organisation focused on user needs and high quality outputs. The Steering Committee has already embarked upon the preparation of a Strategic Plan and goals for the future. Performance measures against which MoU signatories, the standards organisations and industry can judge the success of our activities will form an important aspect of the Plan. We are also working to produce a Best Practice guide for TWAs (see summary of outline in this issue) in order to capitalise on the wealth of experience already gained by VAMAS.

Protection of the environment is now a major driver for technological change and so VAMAS signed a memorandum of understanding with CTI, the Climate Technology Initiative of OECD/IEA to address environmental issues within the remit of VAMAS. At the joint Steering Committee / TWA Chairmen's meeting held in September 1998 we agreed to apply a co-ordinated approach to examine and define a role for VAMAS in this area (see article in this issue).

At its last meeting, the Steering Committee approved the formation of TWA23 which is concerned with thermal properties of thin ceramic films and the first project will focus on measurement of thermal conductivity. TWA19 on measurement of crack growth in brittle materials has successfully completed its work and the results have been applied to amend an existing standard for ductile materials.

Finally, I would personally like to thank all enthusiasts and supporters of VAMAS. My special thanks go to the TWA Chairmen, who have shown great inspiration and commitment to international co-operation and networking that is at the heart of VAMAS. Without your efforts we would not be in the position of looking ahead to the next millennium as the sole survivor of the measures enacted following the G7 Economic Summit Meeting in Versailles in 1982.

Kamal Hossain
Chairman

• Standards Highlights •

VAMAS fosters the development of internationally acceptable standards for advanced materials by the various existing national, regional, and international standards agencies. A major focus for each Technical Working Area is to further strengthen its ties to the standards-writing community. With the increasing number of concluded pre-standards research projects, it is essential that the results be rapidly transferred to standards-writing organisations. Although not every pre-standards research project produces definitive test results in direct support of a specific standards effort, we continue to see the impact of VAMAS efforts through their recognition in an increasing number of adopted standards.

Standards Highlights identifies draft or adopted standards documents from national, regional, or international standards bodies that are based all or in part on technical outputs from VAMAS TWAs. In the absence of a central standards clearing house, VAMAS participants are strongly encouraged to notify the Secretariat of any such adopted standards and to send a copy to the VAMAS Secretary.

IEC 60050IEV 815	International electrotechnical vocabulary chapter:815 Superconductivity, FDIS; June 1998
	VAMAS contributor - TWA16 Superconductivity, Dr H Wada
IEC 61788-2	Superconductivity, Part 2: Critical Current Measurement, "DC Critical Current of Nb3Cn composite superconductors", FDIS; July 1998
	VAMAS contributor - TWA16 Superconductivity, Dr H Wada
IEC 61788-3	Superconductivity, Part 3: Critical Current Measurement, "DC Critical Current of Ag-Sheathed Bi-2212 and -2223 Oxide Superconductors", CDV; March 1999
	VAMAS contributor - TWA16 Superconductivity, Dr H Wada
ASTM E1457-98	Standard Test Method for Measurement of Creep Crack Growth Rates in Metals
	VAMAS contributor - TWA19 High Temperature Fracture of Brittle Materials, Dr K Nikbin

• Recommendations for Conducting Round Robins •

One of the objectives of the meeting at NPL in September 1998 between the VAMAS Steering Committee and TWA Chairmen was to share ideas to develop best practice for the design and operation of Round Robin intercomparisons. While intercomparisons are invaluable to the pre-standardisation process, embarking on large intercomparisons without careful planning can be an expensive and an unrewarding exercise. Initiation at too early a stage can lead to the generation of a multitude of intractable data, which the organisers feel bound to explain in order to justify the work undertaken. This can lead to a great deal of time being spent analysing what are essentially poor data and reaching conclusions which are generously interspersed with anomalies, buts and maybes. Inevitably the final conclusion is that the work should be repeated with better control of the testing parameters used.

As one of the earliest TWAs in VAMAS, TWA3 on Ceramics, led by George Quinn, has developed, through hard earned experience, some basic guidelines for consideration when setting up a round robin. During the September meeting a number of additional points were made by other experienced TWA chairmen and these have been included with the TWA3 guidelines. While many of the following preliminary criteria may seem obvious, it is too easy to think about them when it is all too late:

1) Ensure that there is a well defined and focused objective,
2) Run a smaller intercomparison initially with a limited number of participants,
3) Keep the work expected from each participant down to a maximum of about one man week,
4) Ensure as far as possible that the material used is consistent and uniform,
5) Give the participants extra material to allow for trials and errors,
6) Ensure that the instructions are written simply and clearly,
7) Do not add too many "interesting" side issues,
8) Start with no fewer that 6-8 participants to allow for a number who will not respond,
9) Make it clear that you will adhere to deadlines for submission of data,
10) Expect that the analysis will probably have to cover issues not originally anticipated,
11) Reports and data release should be made in a timely fashion,
12) All participants should have the opportunity to review and correct a draft version of a final report,
13) Consult a statistician if you are in any doubt about experimental design or treatment of data. It helps to think about this aspect at an early stage.

• New VAMAS Initiative •

Definition of a Role for VAMAS Participation in "Environmental" Standardisation Activities for Materials Technologies

The Steering Committee has approved a proposal to assess the potential for a VAMAS initiative on environmental standardisation activities for materials technologies. The proposal was presented by Sam Schneider from NIST in the USA, who has successfully carried through the work of TWA14 on a classification system for advanced ceramics. Sam Schneider is now following up the proposal with assistance from BAM in Germany, NPL in the UK and colleagues at NIST. Prof Czichos at BAM will provide guidance on behalf of the Steering Committee. A summary of the proposal is given below:

Objective and Justification:

The objective of this proposal is to confirm the need, to define a role and a strategy, and to implement an action plan for VAMAS to address environmental issues confronting materials industries. Currently VAMAS involvement in the environmental arena is minimal. Greater participation by VAMAS should be seriously considered, as its property/performance pre-standards efforts must be allied with companion environmental standardisation activities to assure that "benign" materials products can be processed, manufactured and used at affordable costs.

Importantly, economic and health concerns associated with maintaining stewardship of the environment have led to strategic changes and new policies by national governments. Accordingly, a new paradigm has emerged-pollution avoidance to take precedence over pollution control. VAMAS technical capabilities, standards know-how and breadth of contacts within many countries could provide a tool for a coordinated approach in this area on an international scale.

Background:

Pollution Control

Materials industries contribute significantly to wealth creation, but they also are major contributors to multi-billions of tons of waste generated each year the world-over. Hence, maintaining stewardship of the environment has become a paramount concern in the production and use of commodity and advanced materials. For many years nations have issued mandatory regulations (often linked to health issues) that principally focused on pollution control. "Environmental standards" (really compliance specifications) have been developed by a nation's regulatory agency that set emission/waste limits for a host of industries, from food production to manufacturing of materials.

In parallel with regulatory activities, ISO, in liaison with other standards development organisations, has developed a set of "management" standards (ISO 14000 series) which provide means to improve the environmental performance of organisations and their products and services. Emphasis areas include management systems, auditing, performance evaluation, life cycle assessment, terminology, etc. This "command and control" process has produced cleaner water, cleaner air and unspoiled land, but at substantial costs.

Pollution Avoidance

Pollution control activities no doubt will continue for many years, and effective as it is, different approaches are needed. A new trend "avoidance" has emerged to take precedence over pollution monitoring and assessment, control, remediation and restoration. Avoidance equates to advanced materials designs and processing that yield products benign to the environment. In the main, new or revised (property and performance) standards are needed for materials development in the areas focused on substitution, recycling and reuse.

Action Plan:

Organisation

The first step in the action plan is formation of an ad-hoc VAMAS "Environmental Materials" Group (EMG) comprised of selected steering committee members and TWA chairs knowledgeable about ceramic, metal, and polymer technologies. The primary objective of the EMG is to obtain information from various sources and provide recommendations to the Steering Committee for decision making.

Liaisons and Alliances

Information gathering requires collaboration and assistance from diverse sources. Liaisons and alliances should be established as needed between VAMAS and (national/international) trade associations, professional societies, standard development organisations, government agencies, and selected industrial companies.

Workshops

Conducting a workshop(s) is a necessity, as it will provide primary information needed by the general technical arena regarding pollution avoidance, as well as buy-on/endorsement by key organisations. The workshop(s) might include:

Topic: Pollution Avoidance for Materials Technologies

1. Focus areas: (in priority order)
   a. Substitution
   b. Recycle
   c. Reuse

2. Determination of "technical roadblocks" for each focus area

3. Development of standards needs for each focus area
   (note: includes also processing standards)

4. Sponsors (to be determined)

5. Date/Location (selected to "piggy back" another technical meeting)

6. Summary workshop report to be published as a VAMAS Report

Note: Individual workshops on ceramics, metals and polymers perhaps should be conducted separately.

Final Report to VAMAS Steering Committee

The report will summarize all activities of the EMG and recommendations for VAMAS consideration.

• New Technical Working Area •

Thermal Properties of Thin Ceramic Films

A new Technical Working Area, TWA23, was approved by the Steering Committee at its meeting in September. The objective for the TWA will be to evaluate measurement methods for determining thermal properties of thin ceramic films and coatings and it will address an identified need for standards and codes of practice for thermal measurements on coatings used in important industrial applications.

The technological importance of measuring the thermal properties of thin films has not been fully recognised until recently. Few techniques have been available and the reliability and uncertainty in these techniques has not been established. In the area of thin film thermal conductivity measurement there have been publications that report measurements based on several different techniques. A recent workshop indicated that there is no consensus regarding the relationship between thin film thermal conductivity to bulk thermal conductivity. In order to model systems that depend on the transport of heat through thin films, one must be able to perform measurements of thermal conductivity that minimise measurement uncertainty. While many techniques measure thermal diffusivity, it is important to measure thermal conductivity because this is the property of direct interest.

At the present time there are no existing standards or industrial codes of practice, nor are there any standardisation activities currently in progress, in the area of thin film thermal measurements.

Industries that would benefit from this activity are the semiconductor device industry, the high power laser industry, and industries that rely on coatings for thermal barriers such as the aircraft and the automotive industries.

The plan is to enlist the cooperation of measurement laboratories that are interested in evaluating methods for determining thin film thermal properties. The initial project of technical focus is a round-robin to measure the thermal conductivity a thin films of silicon dioxide on silicon wafers. The thicknesses of the films are in the range 50 nm to 500 nm. Each participating laboratory is to choose its own measurement technique and the results of the measurements are to be compared. There is an interest from ASTM Subcommittee E37.05 on Thermophysical Properties Measurement in the output from this project in order to evaluate the feasibility of standards in thin thermal conductivity measurement.

The TWA Chairman will be Albert Feldman from NIST. Contact details are given on page 31 of this issue.

• Feature Article •

THE CHANGING SCENE FOR ISO

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When ISO was created in 1946, its objective was essentially to provide recommendations to its members aimed at harmonising national standards and for the first twenty five years of its existence, the results of ISO's technical work were published as ISO Recommendations. In all probability, however, very few ISO Recommendations were used during that period as the basis for national standards.

It was only in the early 1970s that ISO began to publish International Standards and with hindsight this was a very opportune moment given the significant expansion of international trade which followed. By the early 1980s, it was starting to be accepted that ISO standards had their own validity in the market place and by the latter part of the decade we saw the first signs of what has come to be known as the globalisation of markets. These were interesting times for ISO and those involved with the organisation. Not only were ISO standards starting to establish their own validity in the market place, but in many instances ISO was being asked to prepare the first standards on particular topics and was consequently moving away from its original mission of harmonising national standards. Global markets would need international standards, it was reasoned, and the future of ISO was therefore assured.

That assurance was to be challenged by two major events in the late 1980s/early 1990s, however. The first was the decision by the European Community to create the Single European Market on the basis of very general, Community-wide legislation supported by voluntary consensus standards developed by the European standards bodies. For many years, the European Commission had accepted that ISO standards could serve for the purposes of harmonisation within Europe and the activities of ISO's counterpart at the European level, CEN, the European Committee for Standardisation, had generally been low-key. This new decision, to establish the so-called New Approach Directives in Europe, resulted in a very significant expansion of European standardisation activity and in many cases a diversion of resources for standardisation from ISO and to CEN, bearing in mind that Europe has always been one of the major contributors to international standardisation. In some instances, therefore, the activities of some ISO standards-developing groups came essentially to a standstill, which, not

unexpectedly, caused some degree of consternation amongst ISO's non-European members. Ultimately, this development resulted in the establishment of the Vienna Agreement on technical cooperation between ISO and CEN, by which both organisations benefit from the results of standardisation work at the international and European levels and also by means of which ISO's non-European members can influence European standardisation. With this has also come the realisation that the Single European Market is not an end in itself and that given the volume of trade to and from Europe the SEM needs to be integrated into the global market. And a final consequence has been a much increased rate of adoption of ISO standards as national standards, not only within the European Union, in which around 40% of all European standards are direct adoptions of ISO standards, but also in other countries around the world.

All well and good? Well, not quite.

The second major event which emerged in the 1990s was the realisation that the traditional hierarchy of international, regional, national and company standards was increasingly being supplemented by "standards" developed in other fora, generally called consortia., particularly in areas of fast-moving technology. Such documents generally represent an agreement between major market players and not the wide consensus which is typical of an ISO standard. Undeniably however such documents were achieving the status of de facto international standards in the market place and in some sectors therefore the question was whether ISO's methodology for reaching agreements on standards was still relevant.

Over the last few years, various consultations have been carried out with industry and it has been confirmed that the activities of consortia do not represent a threat to the formal standardisation system. The vast majority of industries continue to need international standards developed according to the full ISO process of consensus and transparency and in most cases the timeframes required for the development of such standards are acceptable. At the same time, it has to be recognised that the documents developed in the alternative fora are meeting a market need and that generally these are produced more rapidly than ISO standards and therefore are much more able to respond to the rapid product development and marketing cycles which have become typical of some industries.

As part of its commitment to continuous improvement of the ISO process, therefore, ISO engaged in a number of studies to provide streamlined procedures by which ISO can respond to particular market needs for normative documents developed on short timescales. The result of these studies introduced procedural options which can be used at the discretion of those ISO technical committees for which speed of standards development is a paramount consideration, and by which two new types of normative documents, representing reduced levels of transparency and consensus,but which seem to respond to market requirements in at least some sectors, can be produced.

These streamlined procedures recognise that in the elaboration of an ISO International Standard, three different levels of consensus are achieved. The first level comprises a consensus between individual technical experts in an ISO working group, during what is called the preparatory stage in the ISO process. Once this consensus has been attained, the next part of the ISO process involves the formulation of national consensus positions and their negotiation in the ISO technical committee or subcommittee until an international consensus is reached. Finally, this consensus is then exposed to the full ISO membership and in many countries national public reviews are carried out to ensure that by the time the final text of an International Standard is agreed, any party potentially interested in or affected by a standard, has had the opportunity to contribute and make their comments known.

In summary, therefore, ISO has agreed to make available normative documents which represent one of these three levels of consensus. A committee will therefore in the future have the option, once agreement has been reached between the technical experts, either to circulate the document to initiate the national consensus building process OR to publish the text as an ISO Publicly Available Specification. Agreement to publish a PAS will comprise approval by 50% of the members voting of the responsible committee. Similarly, at the end of the national consensus building phase, once the international consensus has been attained, a committee will have the option of either forwarding the text for further processing as a draft International Standard OR requesting that it be published as an ISO Technical Specification. Agreement in this case requires approval by 2/3 of the members voting of the committee. Texts which have been through the full process will continue to be issued as International Standards.

Committees will also have the option of adopting normative documents developed outside the formal standardisation system and according to the case process them for publication either as ISO Publicly Available Specifications, Technical Specifications or International Standards.

The opportunity has also been taken to rationalise the deliverables which for many years have been issued as ISO Technical Reports. These were essentially of three types: Type 1 for documents which had been intended to become standards but for which the required levels of agreement could not be attained, Type 2 to describe either the directions of standardisation in particular fields or in some instances to make available an experimental standard for trial use, and Type 3 which was for information only. In future, the term technical report will be retained purely for informative documents and Type 1 and Type 2 technical reports will be published as Technical Specifications.

In a related move, the ISO Council has decided to add another mechanism to ISO's armoury for providing normative documents which will not rely on the customary technical committee structures. Essentially this will be through an open workshop mechanism whereby market players will be able to negotiate in a workshop setting the

contents of particular normative documents. The results of such workshops would lead to the publication of documents designated as Industry Technical Agreements.

The procedures and designations of these new deliverables have been agreed between ISO and its partner in the electrotechnical field, the International Electrotechnical Commission, IEC. Attempts are now being initiated to seek the same harmonisation between ISO/IEC and the European standards bodies, CEN, CENELEC and ETSI.

ISO will continue to offer to VAMAS the possibility of publishing the results of research as Technology Trend Assessments, but there may be instances in which a VAMAS TWA might wish to propose that the output of a TWA should be progressed for publication as a PAS or as a Technical Specification. The important difference here is that a TTA will remain on ISO's books until such time as it is either superseded by a normative document developed through ISO's committee structures or the organisation is advised to withdraw the TTA. On the other hand, PAS and Technical Specifications will have a maximum life of six years and at the end of this period they will need either to be processed to become International Standards or be withdrawn.

As noted in the introductory paragraphs, ISO's role has evolved over the first fifty years of its existence and will no doubt continue to do so in the future. The environment for which ISO seeks to provide its services is constantly evolving so it is not unnatural that the organisation needs to be responsive to newly emerging requirements for standards and related documents to be used by market players and the economic partners of the voluntary standardisation system. We hope that the new range of deliverables will respond to current market requirements. If it allows strengthening of ISO's links with the world of research, that will be an added bonus.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 30
Efficient test procedures for polymer properties
Warren D Ketola and Richard M Fischer
3M Centre, St Paul, MN 55144-1000, USA

Objective: To define a procedure that can be developed into a standard for selecting and evaluating any type of reference material to be used in an exposure test.

A series of round-robin exposures of reference materials was undertaken jointly by the ASTM G3 Committee and VAMAS TWA12. Three separate accelerated durability round-robin studies were conducted. In the first, polysulfone films were exposed to nearly monochromatic UV light and the change in absorbance at 330 nm was measured. In the second, polystyrene plaques in water-cooled xenon arcs were submitted to a range of radiant exposures. In the third study, polyethylene films were exposed in filtered open flame carbon-arc devices. Using transmission IR spectroscopy, the carbonyl index of each specimen was calculated to give an indication of the oxidation of the film.

In each study, participating laboratories were asked to make the sample measurements using two operators working independently of one another so that the repeatability of the property measurement could be evaluated. The samples were then returned to the reference laboratory where properties were measured again by one person using a single piece of equipment.

For each round robin, an analysis of variance was conducted to determine the contribution of the exposure, property measurement, and the reference material to total variability. Repeatability and reproducibility limits were also determined for the data from each round robin. In all three round robin studies, the primary source of variability was found to be the exposure itself. The differences between replicate specimens of the reference material and between operators contributed little to overall variability for exposures of polysulfone and polystyrene. There was a great deal of variability in results for the round robin exposures of the polyethylene film. While the exposure itself was the major source of variability, the effects of exposure position and variability in the measurement of carbonyl index by different operators could not be ignored.

Analysis of data from a round-robin study conducted by the IFAI (Industrial Fabrics Association International), which evaluated exposure of 24 test fabrics and four reference materials, showed that good between-lab agreement for a reference material often does not translate to good between-lab reproducibility for test materials. There

was poor reproducibility in absolute colour change reported by the labs, even amongst those that produced nearly identical results for the reference materials. However, if asked to rank the colour change of the 24 test fabrics against a control fabric, the labs showed excellent agreement. Ranking performance of test materials compared to control materials exposed at the same time is more effective than the use of reference materials for coping with the variability inherent in all exposure tests.

Based on the results of this research, the ASTM G3 Committee on Weathering and Durability are developing standards for characterising the repeatability and reproducibility limits for reference materials used in round robin tests.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 31
Bend strength measurements for hardmetals. International prestandardisation collaborative activity Part 2 Analysis of results

B Roebuck
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Objective: To examine the effect of different testpiece geometries on the bend strengths of hardmetals. The project was devised to take account of developments in the understanding of strength measurements, the increasing desire for data more relevant to material quality and design, and to work towards a test that can give results comparable to test data obtained on competing materials, such as ceramics and cermets.

A round robin study involved 14 laboratories, mostly industrial, in eight countries, testing seven materials to 11 testpiece geometries. The main purpose of the exercise was to examine the effects on strength of testpiece geometries which are different from the rectangular bars allowed in the current ISO 5227 standard. The alternatives included round and notches testpieces in both 3-pt and 4-pt bend configurations. The full set of results of the tests were reported in a previous VAMAS publication (VAMAS Technical Report No 22, June 1996) and the current report provides the analysis of the results. The hardmetals tested included ultra-fine, fine, medium and coarse WC/Co, a medium-fine WC/ Cubic Carbide/Co and a Ti(CN) based cermet.

Considerable differences in strength values (by up to a factor of two) were obtained from different geometries, although there was good reproducibility in the results obtained by the different organisations taking part. Analysis of the representative strength values and Weibull moduli indicated that the effects of wedging stresses and friction, caused by the inability of the support rollers to rotate freely, were the most likely source of differences in strength observed between the different geometries.

Surface preparation had a very significant effect on strength, particularly for annealing which reduced strength. The strength reduction caused by annealing depends on the material type and geometry. This has important consequences for standardisation, and further systematic work on the effects of surface preparation is required.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 32
VAMAS round robin on fracture toughness measurement
of ceramic matrix composites
Mineo Mizuno and Jun-ichi Kon
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Objective: To compare three different fracture toughness test methods at room temperature by means of a round robin study. Also to investigate the notch tip radius and saw-cut width dependence of fracture toughness. To establish which techniques have potential for use in standards.

Fifteen laboratories in seven countries participated in the round robin on fracture toughness of silicon carbide whisker reinforced silicon nitride. Defects were introduced to the material using three different techniques: pop-in crack, saw-cut, and V-notch.

The fracture toughness at room temperature was measured by three methods; single edged precracked beam (SEPB), single edged notched beam (SENB), and the single edge V-notched beam (SEVNB). The values of fracture toughness, K_{IC} (obtained by the SEPB method) and $K_{c,n}$ (by the SENB and SEVNB methods) were measured with four different flexural modes: 3-pt with a 16mm span, 3-pt with a 30mm span, 3-pt with a 40mm span, and 4-pt with 20 and 40mm spans.

The study found that fracture toughness of ceramic composites can be measured by the SEPB, SENB, and SEVNB methods. No obvious effect of flexure mode on the fracture toughness is observed in the SEPB and SEVNB methods, except for the SEPB 3-pt flexure mode with 16 mm span, which gives a slightly higher value than the others. The $K_{c,n}$ values show saw-cut width or V-notch tip radius dependence, with the value of $K_{c,n}$ increasing with increasing half width of saw-cut or the tip radius.

The SEPB technique produces very accurate measurements of fracture toughness, with an average value of K_{IC} of 6.8 MPa $m^{0.5}$. However, some labs find the technique difficult to use because it requires the introduction of a valid precrack into a specimen before the flexure test is performed. The precrack length and propagating direction have to conform to a defined specification. The success of the technique depends on the skilfulness of the operators. Accurate jigs and some experience are required to precrack the SEPB specimens reliably.

The $K_{c,n}$ values by the SEVNB method show large scatter when the tip radius is more than 30 μm, but small scatter when it is less than 20 μm. Values in the range 6-7 MPa $m^{0.5}$ were achieved, similar to those obtained using SEPB. A notch tip radius of less than 30 μm is therefore recommended to give consistent results.

The values of fracture toughness obtained using the SENB technique show a wider scatter then the other two techniques, with $K_{c,n}$ values ranging from 6 to 13 MPa $m^{0.5}$. This is thought to be due to the differences in saw-cut shape and machinery conditions among the labs, and means that the SENB technique is unsuitable for use in standards without the specification of precise machining conditions for introducing a saw-cut into a specimen.

The SEPB and SEVNB methods are recommended for the measurement of fracture toughness in ceramics. SEVNB has an advantage in that precracking is not necessary. However, precise V-notch machining is required and a tip radius of less than 30 μm is recommended. The results obtained by this study are expected to contribute to future standardisation.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 33
Round robin on recording hardness
Christian Ullner
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George Quinn
National Institute of Standards and Technology Gaithersburg, MD 20899, USA

Objective: To compare depth sensing hardness measurements made on two materials by a number of different laboratories using very stiff hardness machines up to 10 N and commercial devices measuring at forces up to 1 N.

The recording hardness (depth sensing hardness test or continuous indentation test) was measured in a round robin of 11 laboratories at 1 N (9 labs) and 10 N (6 labs) on two materials, optical glass BK7 and a dense silicon nitride ceramic. The raw data of force F and displacement h from each laboratory were analysed by the authors and the hardness parameters were calculated for $F/F_{max} = 0.1$, 0.43, and 1. While the hardness HU based on the penetration depth fluctuated, due mainly to the extrapolation for zero points, the hardness HUs based on the slope of the F versus h plot was more sensitive to inhomogeneities of material and geometry. There was also a correlation between the measured hardness values and the different initial unloading slopes.

The report concludes that both of the materials used were appropriate for use as reference materials because the repeatability of results within the laboratories was good, particularly for the 1 N measurements. Lack of reproducibility between laboratories, particularly for the 10 N tests, was an indication that the measuring procedure needs to take account of different machine stiffnesses to improve the measurement of the indenter displacement relative to the specimen surface.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 34
VAMAS round robin on conventional hardness testing of whisker reinforced ceramic composites

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Objective: To investigate the potential of using the conventional Vickers and Knoop test methods for measuring hardness of a whisker-reinforced ceramic composite through a round robin study involving 21 laboratories from 9 countries.

Two different test forces were used for each technique on silicon nitride ceramics containing 0 vol% (monolithic), 10 vol%, and 20 vol% silicon carbide whiskers. To evaluate whether the Knoop test would show anisotropy, the test material was indented on two different surfaces. On the side surface two test directions, parallel and perpendicular to the length of the specimen, were tested.

Hardness values increased with the increase of silicon carbide whisker content for both Vickers and Knoop tests. The scattering of data was not very different for monolithic or composite specimens, demonstrating that conventional Vickers and Knoop tests are useful and meaningful for testing whisker-reinforced composites.

A fixed test force is recommended for standard hardness tests. Similar hardness values were achieved with the Knoop test irrespective of the surface tested or the indent direction. The surface and direction have little bearing on results and any differences in hardness are within the error bar resulting from the scatter.

The study confirms that Vickers and Knoop hardness tests are useful for the characterisation of whisker-reinforced ceramics as well as monolithic ceramics. It recommends the use of higher test forces than currently proposed in draft standards to minimise scattering. A test force of 10 kgf for Vickers (HV10) is suggested as well as 1 kgf. For the Knoop test a force of 2 kgf (HK2) rather than 1 kgf.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 35
CEN/VAMAS phase volume fraction round robin
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Objectives: To reveal the scatter that occurs in the determination of the volume fraction of phases and/or the porosity in multiphase advanced ceramics, and thus to provide a possible confidence level that can be used in the citation of results. It can also be used to determine the reliability and reproducibility of the results of tests on materials of increasing complexity.

This report describes the results of a round robin conducted under the auspices of CEN Technical Committee 184, Advanced Technical Ceramics. Twenty seven labs took part in the study to evaluate the potential accuracy and repeatability of making manual or automatic image analysis (AIA) characterisations of phase volume fraction in multiphase ceramic materials.

Participants were provided with a series of micrographs of multiphase ceramics of differing degrees of difficulty of interpretation. The quality of the micrographs ranged from an idealised computer-generated microstructure, with a clear delineation of grain boundaries and good contrast between the phases, to backscattered and secondary electron images, where there was less distinction between phases, the presence of pores, and/or a certain degree of noise.

The micrographs were analysed both manually, using a grid intersection counting method, and automatically using a grey level pixel counting method. The results of the manual technique were outstandingly consistent for all micrographs supplied, and provide a suitable basis for the preparation of a CEN standard. The scatter observed in the manual results is mainly due to positioning of the grid and to the distribution of the phases. In contrast, the results obtained using AIA, though statistically consistent and reproducible, were less reliable and more widely scattered. The larger scatter is mainly due to difficulties in differentiating phases by distinguishing and partitioning grey levels of the digitised image. Another factor influencing the scatter is resizing of the image, converting it to another format, or using only a part of the area of the micrograph.

The second part of the study involved providing each lab with an alumina/zirconia test

sample (composed of the same material as used for some of the supplied micrographs) from which a further micrograph was to be prepared using the lab's own methods. The preparation involved as a minimum grinding and etching, and was designed to evaluate the participants' abilities to prepare micrographs of suitable quality. If this was successful, a comparison would be made between the micrograph produced by the labs and the supplied micrographs. The results of both manual and automatic image analysis on the supplied test sample showed a wider scatter compared with the results on the supplied micrograph. This was thought to be largely due to inhomogeneity of the sample.

Technical Working Area 13

LOW CYCLE FATIGUE

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Objectives

- To develop a framework for quantifying measurement uncertainties in low cycle lifetime data.
- To validate the new Code of Practice for the measurement of bending caused by load misalignment in uniaxial fatigue testing.
- To provide recommendations for a best practice for routine testing of metallic materials.
- To provide pre-cursor information for the development of an LCF reference material.

The VAMAS/EC project on "Quantifying Data Uncertainties and the Validation of a Code of Practice for the Measurement of Bending in Uniaxial Fatigue Testpieces" finished in September 1998. The main objectives of this project included the development of a good practice testing procedure that would reduce variability of inter-laboratory lifetime data to within a factor of five (in the previous VAMAS/EC project, factors of up to 60 were observed) and to validate a new Code of Practice on machine alignment measurement.

More than forty inter-laboratory LCF tests at ambient temperature were carried out by five laboratories on specimens that were machined in one laboratory and made of the same batch of Nimonic 101 superalloy material. Special care was taken to align all the test facilities involved so that the maximum bending strain due to misalignment of the test system was below 50 micro strain or 5% of the applied axial strain. Two participants had slightly higher bending than allowed but were within 10%. The fatigue test results showed remarkable agreement between laboratories so that the scatter in lifetime (N_{fmax}/N_{fmin}) was less than a factor of two.

A further 30 tests were carried out at 850°C. Only three laboratories were involved in this part of the work and the resultant variability in lifetimes was within a factor of six. Although the numbers of participants in the recent series of round robins are fewer than in the previous project, the recent results indicate an order of magnitude improvement on the earlier results. In addition, eighteen tests at 850°C were carried out by one laboratory on one machine with predetermined levels of bending ranging from 2 to 40%in order to establish the effects of bending on the material's endurance behaviour. These tests have indicated that as bending increases, lifetime significantly decreases.

More than fifty sets of inter-laboratory machine alignment and specimen bending measurement tests were performed using strain-gauged specimens made of either Nimonic 101 or aluminium alloy 6063. These tests were conducted as part of the validation exercise of the "Code of Practice for the Measurement of Bending in Uniaxial Low Cycle Fatigue Testing". Each specimen was equipped with either two or three sets of gauges, four gauges per set. Some bending measurements were performed under plastic conditions. In order to investigate the effect of specimen size on machine alignment measurement, a further number of tests were carried out on a range of specimens, all having the same parallel length but with different diameters. It was surprising that in one round robin measurement exercise that involved eight organisations using aluminium specimens, none was able to meet the bending limits recommended in the new Code of Practice and other national and international standards. Subsequent investigation showed that the reason was in fact due to high levels of "inherent bending" in the test specimen themselves rather than due to misalignments in the test systems. A report on this was produced and as a result the new Code of Practice is being revised.

A major problem was to analyse, systematically and reliably, the results of the bending measurement tests. To this end, two prototype software programs called ALIGNCAL and BENDCAL were developed and validated. ALIGNCAL separates the bending contribution due to the machine misalignment from that due to errors inherent in the specimen itself and gives a classification of the machine alignment. The programs run on Microsoft Excel Version 5.0 or higher, in Windows 3.1 or 95 environment. The final programs will be made available to the TWA members.

A number of recommendations from this work have been disseminated to the ISO/TC164/SC5 meetings held at DLR Institut für Werkstoff-Forschung, Cologne, on 17-19 September 1997 and at Cape Cod, Massachusetts on 3-4 September 1998. Three draft international standards on low cycle fatigue are now known to have adopted the new Code of Practice for specimen bending measurement. These are ISO TC164/N81, PrEN 3874 and PrEN 3988.

A presentation on 'The Latest VAMAS Developments in Alignment Measurement' was given to the ASTM E28.04.03 Workshop on Alignment that was held in Norfolk, Virginia on 4 November 1998. The ASTM is currently revising its ASTM E1012 Standard. It is intended to continue and strengthen the current

dialogue and exchange of information and experiences between TWA13 to the ASTM Group under the chairmanship of Dr James Hartman.

Technical Working Area 16

SUPERCONDUCTING MATERIALS

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Objective

- To establish reliable measurement techniques for high temperature superconducting materials through the use of round robins on critical current, critical temperature, and critical field measurement methods.

This article briefly describes the activities that have taken place within VAMAS on superconducting materials since the formation in 1986 of TWA6, which included work on cryogenic structural materials in addition to superconducting materials. Measurement methods for practical metallic superconductors were investigated in TWA6 but in 1993 the TWA was split in recognition of the different techniques used for electrical and mechanical measurements. Work on superconducting has since been undertaken within TWA16 and work on mechanical measurements for cryogenic structural materials has been undertaken within TWA17. The activities of the new TWA16 now extend to the characterisation and evaluation of new classes of superconductors, including oxide superconductors.

Projects Implemented

1. DC critical current measurement method for Nb_3Sn

Two series of interlaboratory comparisons of critical current measurements were carried out on Nb_3Sn multifilamentary conductors. Twenty four organisations from Europe, Japan and North America took part in the first intercomparison and thirteen organisations participated in the second, where each participant was supplied with a specimen of a standard NbTi (NIST-SRM 1457) conductor as reference material.

In 1995 a TWA report on these intercomparisons and complimentary studies was published as a supplement volume of Cryogenics journal [1]. This report included a glossary of superconductivity and cryogenics and a recommended dc critical current

measurement method that were both adopted as the base for corresponding IEC (International Electrotechnical Commission) standards.

2. AC loss measurement method for NbTi

Two series of intercomparisons of ac loss measurements were carried out on NbTi multifilamentary conductors, involving a total of thirty-one organisations. In 1997 two reports on these intercomparisons were published in Cryogenics [2,3]. In one of the reports where dc magnetisation measurements were reported a guideline was proposed for hysteresis loss measurement using a VSM (Vibrating Sample Magnetometer) [2]. This guideline is now being considered as the base for a corresponding IEC standard.

3. Critical field measurement method for NbTi

A measurement intercomparison on the upper critical field in NbTi was implemented with the participation of 12 laboratories. NIST-SRM 1457 NbTi wires were used as sample conductors. The results were reported in IEEE Transactions in 1995 [4].

4. DC critical current measurement method for Ag sheathed Bi-oxide superconductors

An independent critical current measurement intercomparison was carried out on short Ag-sheathed Bi-2212 and/or -2223 oxide superconductor tape samples in the USA (6 participants), Europe (11 participants), and Japan (8 participants). A report based on these intercomparisons is in preparation for publication in Cryogenics. In September 1997 TWA16 prepared a "guideline for a dc critical current test method for Ag-sheathed Bi-2212 and -2223 oxide superconductors" which will be included in the report. The guideline was transferred to IEC/TC90 and is now proceeding towards an international standard.

Projects Proposed

Since 1997 activities of TWA 16 have become more extensive by involving researchers interested in bulk and thin-film oxide superconductors. Thus, there are now three sub-working areas in TWA16; wires and tapes, bulk and thin-films. In each of these sub-working areas, the following projects have been proposed:

Wires and Tapes

- Strain effect measurement methods for metallic and oxide superconductors.
- AC loss measurements on new classes of superconductors.
- Evaluation of mechanical and thermal properties of oxide superconductors.

Bulk

Development of a standard measurement method for levitation forces.

Thin-films

Development of a standard measurement method for surface resistivity.

Links with IEC TC90

There is a strong synergy between the work of VAMAS and Working Groups on wire and tape, thin films and bulk properties in IEC TC90. Activities in bulk and thin layers are directly oriented toward the development of IEC standards. Thus, the work areas of TWA16 are closely related to the standardisation needs of IEC/TC90.

Summary

Activities and outputs are summarized below:

Project	Publication		IEC Status
critical current in Nb_3Sn	VAMAS supplement	Guideline	FDIS/IEC/TC90 (IEC 61788-2)
		Glossary	CDV/IEC/TC90 (IEV chapt. 815)
critical field in NbTi	TWA report		
ac losses in NbTi	TWA reports	Guideline	NP/IEC/TC90
critical current in Bi-oxides	VAMAS supplement (in preparation)	Guideline	WD/IEC/TC90 (IEC 61788-3)
ac losses in Bi-oxides (proposed)			
strain-effect (proposed)			
levitation force (proposed)			
surface resistivity (initiated)			WD/IEC/TC90 (IEC 61788-7)

References

1. Wada H, Goodrich LF, Walters C, Tachikawa K (eds) (1995) Cryogenics 35-VAMAS Supplement, Elsevier Science, UK
2. Collings EW, Sumption MD, Itoh K, Wada H, Tachikawa K (1997) Cryogenics 35: 49-
3. Schmidt C, Itoh K, Wada H (1997) Cryogenics 35: 77-
4. Tachikawa K, Koyama S, Takahashi S, Itoh K (1995) IEEE Trans. Appl. Supercond. 5: 536-539

Technical Working Area 20

MEASUREMENT OF RESIDUAL STRESS

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Objectives

- Establish accurate and reliable procedures for making non-destructive residual stress measurements by neutron diffraction.
- Examine samples in which residual stresses have been introduced by different techniques.
- Conduct inter-laboratory comparisons to establish reproducibility.
- Assemble the necessary information for preparing a draft standard for making the measurements.

The first project in this TWA is concerned with the measurement of residual stress using neutron diffraction. This method is currently the only technique available for non-destructive measurement of residual stress deep inside components, due to the high penetration depths of neutron beams. There is wide interest throughout the world in exploiting this technique to examine relatively large components where the consequences of unexpected service failures could be catastrophic. The work being undertaken by VAMAS is concerned with the establishment of calibration methods and procedures and forms part of a large international effort to establish this technique as a standard method available for use by industry.

The inaugural meeting was held at the ILL, Grenoble, France in January 1996. At this meeting 5 Task Groups were set up to address:

- experimental protocol
- sample characterisation
- reliability

- comparison and verification
- conversion of strain to stress

It was agreed that the following four types of sample would be examined for a Round Robin inter-comparison: an aluminium alloy shrink-fit ring and plug; a shot-peened surface; a ceramic composite, and a ferritic steel weldment.

Further meetings have been held at AECL, Chalk River, Canada in October 1996, Studsvik, Nykoping, Sweden in June 1997, NIST, Gaithersburg, USA, in May 1998, ORNL, Oak Ridge, USA in November 1998. It is anticipated that the last meeting will take place at ISIS, Rutherford, UK, in July 2000 to coincide with the 6th International Conference on Residual Stress (ICRS-6).

Measurements have been completed on the ring and plug sample and the results analysed. It is intended that the findings will be issued as a VAMAS Technical Report. Measurements on the ceramic matrix composite (CMC) and shot-peened plate are in progress and agreement has been reached on the geometry of steel weldment to be investigated.

A start has been made on preparing a framework for the draft standard. A similar format to that in ASTM E 1426-94 for residual stress measurement by X-rays is being adopted. The route into standards bodies has still to be confirmed.

The group initially consisted of 19 participants, mainly from organisations with neutron sources, but has expanded to about 50 with a growing interest from industry. Communication within the group is through an e-mail reflector to which only members have access. A www site (http://www.risoe.dk/vamas-twa-20/) with a link to the VAMAS site, has been set up for communication with the outside world.

A database of references is being collected together. Several papers have been presented in journals and conference proceedings. Meeting reports have appeared in Neutron News and many presentations have been made describing the potential of the technique. Several neutron sources throughout the world have already installed diffractometers dedicated to stress measurement.

• VAMAS Calendar •

TWA 2 (Surface Analysis)
8th European Conference on Applications of Surface and
Interface Analysis, Hotel Melia-Sevilla, Seville, Spain .....4-8 Oct 1999

TWA3 (Ceramics) Meeting at European Ceramic Society
Conference, Brighton, UK .....21 June 1999

TWA5 (Polymer Composites) Meeting at 12th International
Conference on Composite Materials, Paris, France .....5-9 July 1999

TWA13 (Low Cycle Fatigue)

TWA15 (Metal Matrix Composites) Meeting at 12th International
Conference on Composite Materials .....5-9 July 1999

TWA16 (Superconducting Materials) Meeting at International
Superconductivity Symposium (ISS99), Morioka City, Japan .....16-19 Oct 1999

TWA17 (Cryogenic Structural Materials) Meeting at International
Cryogenic Material Conference, Montreal, Quebec, Canada .....12-16 July 1999

TWA19 (High Temperature Fracture of Brittle Materials)
Meeting to discuss objectives for a new activity on test
methods for components containing defects at Imperial College,
London, UK .....25 June 1999

TWA20 (Measurement of Residual Stress) Meeting at
JRC, Petten, Holland .....7-9 July 1999

TWA22 (Mechanical Properties of Thin Films and Coatings)
Meeting at International Conference on Metallurgical Coatings
and Thin Films (ICMCTF99), San Diego, USA .....12-16 April 1999

Anyone wishing to join a meeting should contact the relevant TWA Chairman.

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