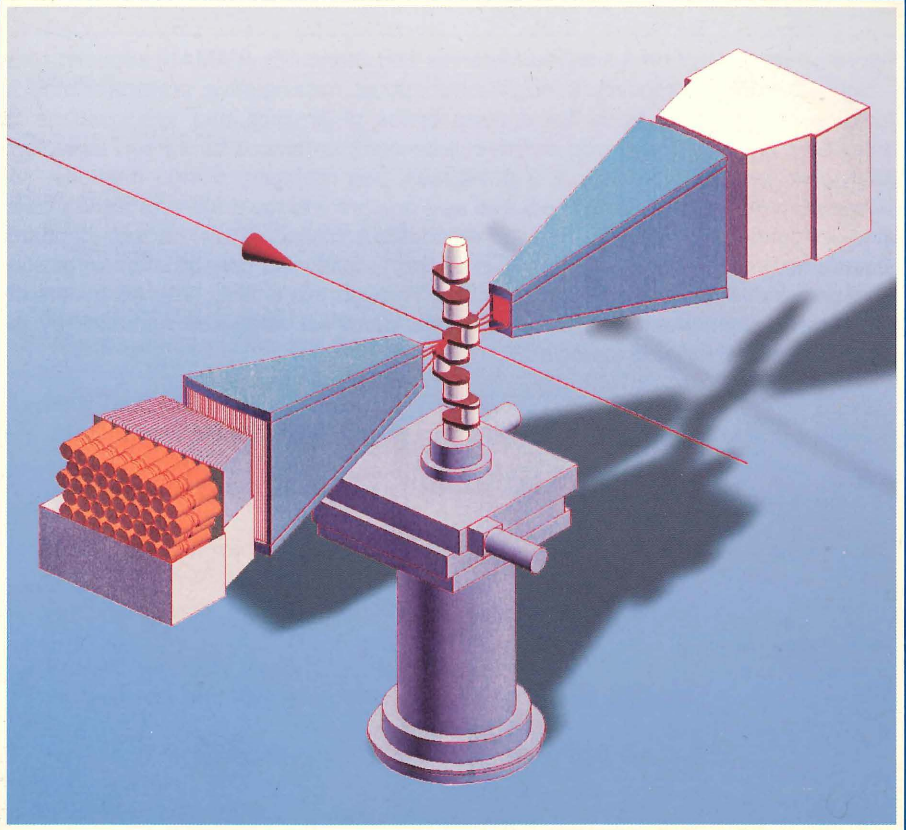
VAMAS



BULLETIN NO. 20
July 1996

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 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.

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Cover: Schematic representation of a crank shaft in the neutron beam of an instrument called ENGIN which has been specially developed at the Rutherford Appleton Laboratory (RAL) for neutron strain scanning. The technique can be employed for determining residual stress distributions non-destructively in the interior of crystalline solids

Illustration courtesy of Rutherford Appleton Laboratory, Chilton, Oxfordshire, United Kingdom

VAMAS

• Table of Contents •

• Foreword •

It is a pleasure for me to take on the role of chairman of VAMAS for a second term following the three year tenure of Harry Rook from NIST in United States. As many of you are aware I am the Director of the Centre for Materials Measurement and Technology (formerly the Division of Materials Metrology) at the National Physical Laboratory in the United Kingdom. During this period of my Chairmanship, the VAMAS Secretary will be John Sillwood who has been involved with VAMAS activities in the UK over the past three years.

We are indebted to Harry Rook and to Jim Early for their dedication as Chairman and Secretary of VAMAS. During their period at the helm, VAMAS has made real and significant progress. An important milestone has been the signing of the IEC Memorandum of Understanding that now stands alongside the existing Agreement with ISO. Indeed the first joint publication under the IEC/VAMAS Agreement has already been approved. Harry Rook has stepped down from the Steering Committee as he has moved to a new post within NIST and will be replaced by Steve Freiman who is Chief of the Ceramics Division at NIST. Our grateful thanks go to NIST for the provision of the Secretariat and their continued support. I would also like to thank Atsushi Oguchi from NRIM, a long serving Steering Committee member for Japan, for his valuable contributions over the years. On behalf of VAMAS we wish him a long and happy retirement.

This Bulletin has been brought together and edited by Jim Early. Looking back on recent issues, the inclusion of sections on Standards Highlights and Recent VAMAS Outputs have served as a focus on the key mechanisms by which VAMAS presents itself to the world at large. It is pleasing to see that the results of VAMAS are having ever increasing influence in the formulation of national and international standards. Dissemination of the work of VAMAS is being pursued with increased vigour and as part of this process we will be introducing VAMAS pages on the Worldwide Web early in the autumn.

VAMAS will focus more directly on future standards needs to assist international trade and to improve product quality and reliability. Hence closer links with ISO and IEC will enable improved coordination between VAMAS and the standardisers. Where standards committees encounter an urgent problem during the development of a standard, VAMAS should be able to help by using its extensive network of international researchers. An important task during the next twelve months is to extend the current MoU for VAMAS beyond 1997. Member countries are most supportive and keen to continue collaborating, and we look forward to building on the recognised strengths and achievements of VAMAS.

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 organizations. 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 clearinghouse, VAMAS participants are strongly encouraged to notify the Secretariat of any such adopted standards and to send a copy to the VAMAS Secretary.

Recent adopted standards include:

1] G 118-93, ***Guide for Recommended Format of Sliding Wear Test Data Suitable for Databases,*** adopted in 1993, ASTM Committee G 2, Wear and Erosion. VAMAS contributor - TWA 1, Wear Test Methods, E. Santner, Chairman.

2] JIS R1607, ***Testing Methods for Fracture Toughness of High Performance Ceramics,*** adopted in 1990, Japanese Industrial Standards Committee. VAMAS Contributor - TWA 3, Ceramics, G. Quinn, Chairman.

3] C 1322-96, ***Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics,*** adopted in 1996, ASTM Committee C 28, Advanced Ceramics. VAMAS Contributor - TWA 3, Ceramics, G. Quinn, Chairman.

4] C 1326-96, ***Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics,*** adopted in 1996, ASTM Committee C 28, Advanced Ceramics. VAMAS Contributor - TWA 3, Ceramics, G. Quinn, Chairman.

5] C 1327-96, ***Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics,*** adopted in 1996, ASTM Committee C 28, Advanced Ceramics. VAMAS Contributor - TWA 3, Ceramics, G. Quinn, Chairman.

• Feature Article •

NEW TECHNICAL WORKING AREA
TWA 19 High Temperature Fracture of Brittle Materials

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BACKGROUND

The awareness of the limitation of our natural resources and the increasing pressure to control the emission of carbon dioxide leads to the necessity to use more fuel efficient technology. An important measure in this context is the increase of the operating temperature in power plants and combustion engines. This, together with higher design stresses increases the risk of failure under creep conditions. The most advance and hence most dangerous stage of creep failure is given by the propagation of cracks under creep conditions. This was the subject of the activities of the previous VAMAS TWA 11 on Creep Crack Growth. A method for determining creep crack growth was developed which was adopted by ASTM as E1457-92. Thus the goal of creating a standardized test method was achieved and TWA 11 was terminated.

The conclusions on creep crack growth characterization by TWA 11 were derived from studies on materials which behave in a ductile manner under creep conditions. However, many advanced materials proposed for critical applications at high temperature are characterized by relatively poor ductility in creep conditions and are described as "creep brittle". Materials in this category include steels, aluminum alloys, some superalloys, intermetallics, ceramics, and composites. Typical applications include aerospace, power engineering, and increasingly, petrochemical plants. Thus, engineers are being asked to design with materials which are often described as "unforgiving" because of the tendency to brittleness. Therefore, appropriate data to handle crack initiation and growth and to predict component life are needed.

Currently no standards are available either for test methods to obtain relevant engineering design data or for providing guidance on the use of these data in design and component life prediction.

GOALS

In view of the lack of information relevant to the application of creep brittle materials, TWA 19 was established in October 1993 to fill this gap. The fracture behaviour of advanced creep brittle materials at high temperatures will be evaluated and mechanical test methods for crack growth will be established to assess the fracture life of components. Particular emphasis is being given to:

- **Developing a test method for determining the creep crack growth rate of creep brittle materials**

  This method will address materials exhibiting small scale creep at the crack tip. Materials and test conditions in which crack growth is dominated by environment caused by elevated temperature corrosion are excluded from this test method.

- **Understanding material behavior both in terms of mechanics and micromechanisms**

- **Dissemination of information by open workshops and symposia**

- **Feeding the conclusions into standards-making bodies so that valid test standards can be generated**

It is agreed that the TWA should not lose sight of applications, which means that the applied parameter (e.g C^*) in the structural component should be borne in mind. Since active work in this area would overload the present TWA, the application needs will be considered by workshops for which personalities experienced in that field will be invited.

WORKING MECHANISM

The members of TWA 19 are also members of standards-writing and pre-standards organizations, including American Society for Testing and Materials (ASTM) for the US; European Structural Integrity Society (ESIS) for Europe; and Japan Society for the Promotion of Science (JSPS) for Japan, and will be responsible for ensuring the exploitation of the TWA work by these organizations. Thus, TWA 19 coordinates and complements existing activities underway in Europe, Japan, and the United States.

WORK PLAN

The materials envisaged for testing are:

- Titanium alloy Ti 6242
- Aluminum alloy 2519 T87
- C-Mn steel
- γ-Titanium aluminides TiAl and TiAlCr

This set of materials is regarded as a very positive aspect because the conclusions to be drawn will be representative of a wide range of materials. The creep crack growth testing and evaluation follows ASTM E1457-92 as closely as possible, deviating from that standard only where necessary. Particular emphasis is being placed on the following items:

Crack growth measurement is a matter of concern. Specific techniques will not be prescribed as it is more important to set accuracy requirements; these are related to Δa as ±15% or ±0.15 mm, which ever is greater. Initiation criterion of interest to industry can be defined as 0.5 mm of crack growth. The accuracy of the indirect method used should be checked at small amounts of crack growth, otherwise the definition and determination would be obsolete. It is therefore proposed to include a multiple specimen exercise in the initial phase of the program. It is of particular interest to have tests terminated at about $\Delta a = 0.5$ mm. A still unresolved problem is the treatment of secondary cracks ahead of the main crack and crack deflection. This question requires further research.

Pre-cracking brittle materials can be very difficult. The Japanese group managed to fatigue pre-crack TiAl. However, the use of a sharp notch for testing TiAl will also be envisaged as there is some evidence that the creep crack growth curves are identical for both starting conditions.

Displacement measurements will be carried out as specified in E1457-92. Several parameters for correlating the creep crack growth rate, da/dt, are being looked at to define the most suitable one.

Three meetings have already taken place, and it is believed that at a fourth meeting, in 1996, some items of the test procedure to be developed can be finalized.

• Feature Article •

NEW TECHNICAL WORKING AREA
TWA 20 Measurement of Residual Stress

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This TWA was approved by the VAMAS Steering Committee at its meeting in March 1995. The Committee also agreed that the initial project in this area would concentrate on the neutron diffraction method of residual stress measurement. This article describes the background to the project, its objectives and the programme of work planned. A meeting was held on the 9th and 10th of January 1996, which was hosted by the ILL in Grenoble, France, to inaugurate the activity. The meeting was attended by representatives from Canada, Denmark, France, Germany, Japan, Netherlands, UK and the USA. The next meeting is to be held at AECL, Chalk River, Canada in September 1996.

Background

The presence of residual stresses in engineering components can significantly affect their load carrying capacity and resistance to fracture. Residual stresses can be introduced into components during fabrication and as a result of deformation incurred during use. Examples of manufacturing operations which can lead to residual stresses include welding, forging, heat-treatment, and surface treatment processes.

A quantitative assessment of the influence of residual stresses on the performance of components requires an accurate knowledge of their magnitude and distribution. Several methods are available for determining theses stresses. They include X-ray diffraction, neutron diffraction, hole drilling, sectioning, and magnetic techniques. However, only the neutron diffraction procedure is capable of establishing residual stress distributions throughout the interior of components non-destructively. Although the procedure has been developed to a level of precision where it can be used for this application, further work is needed to determine an agreed protocol for making the measurements. The purpose of this initiative is to carry out a co-ordinated programme of research to establish the most appropriate procedures for obtaining reliable data.

The principle of the neutron diffraction method is similar to the X-ray technique. A neutron beam is traversed through a component and the elastic strain obtained in different orientations at selected sites from measurements of the separation between crystallographic planes. Stresses are then calculated from these strains using elastic analysis methods.

Several reactor and spallation neutron sources with suitable diffractometers are available throughout the world for making residual stress determinations by neutron diffraction. These use a variety of detector systems, sampling volume shapes and sizes, beam alignment procedures, and statistical analysis routines. A plan of research has been outlined for identifying the most suitable procedure, and precautions to adopt, for particular applications.

Objectives

At the inaugural meeting the following objectives were agreed:

1. To establish accurate and reliable procedures for making non-destructive residual stress measurements in crystalline materials by neutron diffraction.

2. To make measurements on single phase and multiphase materials containing residual stresses which have been introduced by a variety of fabrication techniques and in-service loads.

3. To conduct inter-laboratory comparisons to determine the extent to which reproducible results can be obtained.

4. To assemble the necessary technical information for the preparation of a suitable standard for the non-destructive measurement of residual stress by neutron diffraction.

Programme

The programme of research has been separated into five major activities and a Task Group assigned to each. These are associated with specimen selection and characterization, experimental methods, reliability of results, determination of stress from strain, and validation criteria. The ultimate aim of the investigation is to produce a draft Standard or Code of Practice for making reliable residual stress distribution determinations by neutron diffraction.

In order to cover a broad spread of practical applications, it has been decided to examine a selection of specimens to include a uniform microstructure, a non-uniform microstructure such as a weldment, a composite, and a dissimilar material interface. It is intended that specimens containing both steep and shallow stress gradients will be considered.

Experimental methods that will be examined will include type of neutron source (pulsed or reactor), use of a single or multi-neutron detectors, beam alignment procedure, role of collimation, shape and size of neutron sampling volume, choice of crystallographic plane and number and duration of measurements. Since most engineering failures initiate at surfaces, special attention will be placed on obtaining accurate near-surface data where, of necessity, sampling volume shape and size will change.

Different methods of processing data will be considered to establish reliable assessment procedures. These will include data collection, storage and transfer arrangements, routines for fitting neutron peak shapes, statistical analysis techniques, methods of obtaining a stress-free calibration, texture effects, and procedures for mapping stress fields.

In order to assist with validation of the neutron diffraction results, comparisons will be made with other experimental methods (such as X-rays, hole drilling, and sectioning) and with finite element stress analysis calculations. The role of microstresses and techniques for converting strain to stress will also be examined. Another factor that needs to be determined is an agreed nomenclature list for the wide range of new terms that will be introduced.

It is intended that a World Wide Web site will be set up for TWA 20 to provide an update of progress and enable efficient exchange of information between anyone interested. Participation in the project is not restricted to those who attended the inaugural meeting. Anyone wishing to collaborate in invited to join. Feedback on the requirements of industry will be particularly welcome. Current participants include: T. M. Holden, AECL (Canada); T. Lorentzen, RISO (Denmark); A. Hewat, P. Convert, R. Wimpory, ILL (France); A. Lodini, M. Ceretti, LLB (France); H. Priesmeyer, GKSS (Germany); T. Pirling, Darmstadt (Germany); W. Reimers, HMI (Germany); M. Ono, RRI (Japan); A. G. Youtsos, JRC-Petten (Netherlands); M. T. Hutchings, AEA (UK); M. W. Johnson, ISIS (UK); P. J. Webster, Salford (UK); P. Withers, Cambridge (UK); G. A. Webster, Imperial College, (UK); C. R. Hubbard, S. Spooner, ORNL (USA); A. Krawitz, MURR (USA); M. A. M. Bourke, LANSCE (USA); and P. Brand, H. Prask, NIST (USA).

• Feature Article •

NEW TECHNICAL WORKING AREA
TWA 21 Mechanical Measurements for Hardmetals

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1 INTRODUCTION

A new TWA has been established with the broad aim of assessing mechanical property measurements for hardmetals (cemented carbides). The primary objective in the first project is to examine new alternative methods to ISO 3327 (Bend Strength Tests for Hardmetals):

i) to take account of recent developments in understanding of strength tests

ii) to make comparisons with competing materials such as ceramics and cermets

iii) to seek improvements in the tests ability to provide relevant design data through an international prestandardisation collaborative activity.

2 INDUSTRIAL BACKGROUND

Hardmetals and Cermets

Modern tool materials with high productivity enable manufactured goods to be produced at reasonable and ever reducing costs. They obviously make a significant contribution to the great majority of engineering activities. Tool materials have to be strong, hard and wear resistant. Probably the most ubiquitous tool material is a composite based on a metal carbide, WC, and cobalt. It is called hardmetal in Europe and cemented carbide in N America and Japan. Although not as hard as diamond it is much stronger and tougher. It is one of the strongest materials ever made by mankind with a tensile strength of 3-5 kN/mm^2 and a compressive strength of 3-7 kN/mm^2. This compares, for example, with 2-4 kN/mm^2 for the tensile strength of high technology carbon fibres. Hardmetal is made in bulk form by a flexible powder metallurgy route. It is also relatively cheap being less than one twentieth the cost of diamond in tool form. In Japan similar hard materials to WC/Co but based on a carbonitride, Ti(C,N)/Co-Ni, are called cermets and are widely used as cutting tools.

In Europe and North America WC/Co hardmetals, sometimes containing other carbides such as Ti and Ta or coated with wear resistant materials, make up about 80-90% of the insert market. Hardmetal tools have a low price (~£3/insert) and greater strength and resistance to fracture and thermal shock than ceramics, but they start to creep and lose rigidity at > 600 °C. However, there are a great many uses apart from cutting tools for hardmetals and some of the most important are listed in Table 1.

Table 1 Uses for Hardmetal Tools

- Hot rolling of rod for tyre reinforcements, wire, bolts, screws, and fasteners
- Senzimir hardmetal rolls for flat polish on rolled stainless steel
- Drill bits and DIY products
- Cutting of shoe leather, tea leaf, tobacco and paper products
- Hole drilling of electronic parts
- Road planing tools
- Coal mining picks
- Cold heading dies
- Powder press tools
- Woodworking saws and routers
- Compression dies for industrial diamond production
- Drawing dies - bar, tube and wire
- Glass cutters
- Parts for manufacture of tiles and bricks
- Nozzles for cement
- Drills, nozzles, and valve parts for offshore oil production
- Non-magnetic tooling for magnet production
- Dies for medicinal pill production
- Can making tools
- Dies for laminations for electrical motors and transformers
- Laminate/chipboard - furniture forming tools
- Engineering cutting tools

The major world markets for all hardmetal tools (for cutting, mining, forming and wear) comprise about £1.2B (USA), £0.9B (Japan) and £1.2B (Europe). Mining forming and wear applications consume some 40 - 45% of the different types of hardmetals. However, engineering cutting tools are the biggest group. Four main types of materials, are used of which hardmetal is the largest. In Europe and North America the proportions are about:

• Hardmetal (80% of which are coated)	50-55%
• Tool Steels/High Speed Steels (HSS)	40-45%
• Superhards (PCD and CBN) and Ceramics	5%
• Titanium Carbonitride Cermets	0-5%

In Japan cermets take up to 30% of the market with proportionately less of the other types. Total 1992 global sales of hardmetal, high speed steel (HSS), diamond, ceramic, cermet and coated cutting tools approached £2.5B, approximately equally shared between Europe, North America and Eastern Asia (Japan, etc). Cutting tools support world wide activities in the machine tool industry of about £250B.

3 STANDARDS

Hardmetals are widely used throughout industry because of their extremely good combination of strength and toughness. However, there is concern that existing standard test methods for mechanical properties are not providing sufficient relevant information to allow their optimum use in a wide range of products.

Hardmetals and other sintered hard materials are used in a very diverse range of applications. They face a wide array of stress/temperature/environmental combinations and simple correlations of one-parameter tests and service performance are usually not viable. Also, the cost of in-the-field testing for every possible new development of structure or processing route is generally prohibitive. Consequently, laboratory benchmark testing is vital; in particular for the characterisation of structure, but also for mechanical tests as well. Industry and supporting research organisations now use a wide variety of tests, some of which are standardised and of long standing pedigree, for example, density measurements. Others are thought to be relevant, are fairly well understood, but have some limitations, such as bend testing (transverse rupture tests). Finally there are research or in-house methods such as impact and corrosion testing which are not covered by widely agreed procedures. The methods and parameters of interest can be classified as shown in Table 2.

Table 2 Test Methods and Parameters for Hardmetals

Structure
- Composition*
- Grain or phase size
- Microstructure*
- Magnetic
  - Coercivity*
  - Moment/Saturation
- Porosity*
- Surface integrity

Physical
- Density*
- Expansion
- Diffusivity
- Resistivity

Environmental
- Corrosion
- Erosion

Mechanical
- Hardness*
- Strength
  - Bend*
  - Compression*
- Fatigue
- Abrasion*
- Impact
- Residual Stress
- Toughness
  - Indentation Toughness
  - Fracture Toughness
- Stiffness
- Creep
- Hot Hardness

The list is dominated by mechanical requirements, which clearly reflects their importance in general engineering applications. The methods for which there are published standards in place are starred (*). ISO or ASTM are commonly used in

Europe and North America. In Japan many indigenous standards (JIS) have also been developed.

Probably the most important aspect of testing is the characterisation of structure from which all else follows. For industry the primary need is the production of products with consistent properties and for this a hierarchy of test methods are used, mostly concerned with the use of the ISO standards, as shown in Table 3.

Table 3 Hierarchy of Test methods for Hardmetals

Primary:	Microstructure	ISO 4499
	Porosity/C Content	ISO 4505
	Magnetic Coercivity	ISO 3326
	Density	ISO 3369
	Hardness (Vickers)	ISO 3878
	Hardness (Rockwell A)	ASTM B294
	Magnetic Saturation (Moment)	No standard available
	Co Content/Composition	Various ISO standards
Secondary:	Bend Strength	ISO 3227
	Compression Strength	ISO 4506
	Abrasion	ASTM B611
	Fracture Toughness	No standard
	Palmqvist Toughness	No standard

Research/Grade Development:

Mechanical		*Physical*	
Corrosion	No standard	Expansion	No standard
Erosion	No standard	Diffusivity	No standard
Fatigue	No standard	Resistivity	No standard
Impact	No standard	Grain size	No standard
Hot Hardness	No standard		

It is significant that many of the properties which have a strong influence on the performance of hardmetal products such as corrosion, fatigue, impact and high temperature strength and toughness cannot yet be assessed by widely agreed test methods. One of the reasons for this gap is, in some cases, the difficulty in devising appropriate methods to measure these properties. Some of these deficiencies are being tackled by ongoing research, particularly to develop methods for relating structure to corrosion, fatigue and the high temperature properties.

Equally of relevance is the cost of performing these tests. The preparation of suitable testpieces for measuring the mechanical properties of hardmetals and similar hard materials is always expensive because of the necessity to use diamond grinding/lapping procedures and the requirement for stress-free surface preparation. At present probably 5-10% of the production costs are those related to measurement requirements in the finished product. Consequently additional testing for properties additional to those already provided by current standards will only materialise when driven by specific customer/producer agreements supported by good technical arguments which demonstrate a clear relationship between the property of interest and performance. This does not preclude individual companies developing new grades and products from using as wide an array of testing methods as possible in a laboratory environment to fully characterise a new product in the development stage.

4 BEND TESTING

Initially the TWA is to examine bend testing, particularly the effects of different geometries. Five materials categories are to be researched using seven different test geometries as shown in Table 4.

Table 4 Matrix of Test Options and no of testpieces

Materials and Source	**Testpieces**		
	Rectangular (3pt and 4pt)	**Notched** (3pt and 4pt)	**Round** (3pt, 4pt and notched)
WC/Co; 6% Co fine grained HV30-1800-1900 Teledyne Advanced Materials	100	50	50
WC/Co; 11% Co fine/medium grained HV30-1500-1600 Boart RC Kennametal	100	50	50
WC/Cubic Carbide/Co; 16%, Co HV30-1500-1600 Kennametal	100	50	50
Ti(C,N) cermet HV30-1500-1600 Sandvik	100	50	50
WC/Co; 12% Co medium/coarse grained HV30-1000-1200 Sandvik American National Carbide	100	50	50

Several test jigs have been made and will be circulated to the participating organisations for use in the tests. NPL will co-ordinate the testing and organise testpiece preparation. Six industrial organisations are providing the materials at no cost.

There is a current ISO standard for bend tests on hardmetals (ISO 3327). However, it is widely recognised by industry that it has a number of limitations, largely arising from the geometry of the testpiece and the difficulty of interpretation of the results. New or modified methods are required to provide data which can be used for better design and materials specification and which can give results which are more consistent for quality control. The range of industries which use hardmetals cover most engineering activities in the industrialised world, for example wear and mining parts, tools, construction and civil engineering, electronics and IT industries. Members of the ISO committee are participants in the exercise and the TWA Vice-Chair (J J Oakes - Teledyne Advanced Materials, USA) is a member of the ASTM committee.

In North America a military standard MIL-STD-1942A for flexure tests on ceramics has been adopted by the ASTM. In Europe a new CEN standard for bend tests on ceramics is in draft. In Japan there is a relevant standard JIS R 1601. The geometries associated with these standards are summarised below in Table 5.

Table 5 Geometries for Ceramic Bend Test Standards

Standard	**Type**	**Width** b(mm)	**Thickness** h(mm)	**Total span** 3 pt & 4 pt (mm)	**4 pt inner span** (mm)
MIL STD 1942A	A	2	1.5	20	10
≡ ASTM C1161-1990	B	4	3	40	20
	C	8	6	80	40
JIS 1601		3	3	30	10
CEN	A	2	2.5	20	10
	B	4	3	40	20

None of these geometries are compatible with the current hardmetal standard ISO 3327.

Recent correspondence with Kennametal USA highlighted the industrial importance of establishing the relative merits of 3 and 4 pt bend tests. In addition, Sandvik UK have expressed views regarding the usefulness of testing round specimens. A number of organisations are currently using tests on round testpieces because it is an important product form. Preliminary data indicate that there is a reduction in scatter associated with this form of testpiece. Accordingly it would be important to include a number of tests in the current exercise. Also, work at NPL has indicated that notched bend tests can give useful data. Furthermore, conversation with colleagues in Japan, where they test different geometries again, has indicated puzzling data obtained in bend tests on ultra-fine grained hardmetals. Clearly there was considerable scope for examining these issues on an international basis in order to generate a widely agreed test procedure which because of the harmonised approach would provide the basis for validated recommended methods of testing.

Previous work at NPL has shown that the bend strength of hardmetals is determined by two factors:

i) the distribution of large defects (pores etc), greater in size than about 20 μm

ii) the intrinsic strength of the average microstructure, termed limiting strength.

Additional work also showed that the limiting strength can be measured using a notched bend testpiece. This test method has several advantages:

a) The scatter is considerably reduced.

b) Much lower loads are required to break testpieces, ie, less damage to jigs and perhaps less expensive test machines.

c) The strength values obtained are higher (because failures from large defects are excluded).

The notched tests performed to date have been on modified ISO 3327 testpieces (type B), ie 14.5 mm span x 2 mm wide x 5 mm high (3 pt) with a 1 mm deep notch with a 0.5 mm notch radius. It is proposed that the tests planned in this exercise will include 4 pt notched tests and 3 pt tests on different geometry specimens.

5 PARTICIPANTS

A group of over 20 industrial companies has already been formed supported by a number of research centres (see Table 6). A core group of 14 participants (~ 85% from industry) will conduct the first programme on bend tests. The remaining 10 industrial companies who are corresponding members support the activity in the first project.

The core group comprises: UK 6; Europe 4; USA 3; S Africa 1

The supporting group contains: UK 4; Europe 3; USA 3

together with the Japanese Trade Association representing many companies in Japan. The industrial contribution is the supply of materials (at no costs), participation in meetings (planning already taken place) and conducting tests (primarily by industry, 12 of the 14 testing organisations are industrial).

7 DISSEMINATION

Interim progress reports are discussed at quarterly meetings of the British Hardmetal Association Research Group which contains members from 12 of the participating group including representatives of large multi-national organisations, and medium and small engineering companies.

Informal planning and progress meetings have also been held at appropriate international conferences:

Reutte, Austria, 1993 *Plansee Seminar*	Bonn, Germany, 1994 *Advances in Hard Materials Production*

Maui, Hawaii, 1995
International Conference on the Science of Hard Materials

with a next meeting planned for Stockholm, Sweden (Advances in Hard Materials Production) in May 1996. The TWA activities have also been tabled at recent appropriate ISO committee meetings.

Table 6 Participants

Organisation	Material Supplier	Organisation	Material Supplier
NPL (UK)		Metallwerk Plansee (Austria)	
Dymet(UK)		British HMA	
Sandvik (Sweden)	Yes	Fansteel (USA)	
Sandvik HM (UK)	Yes	General Carbide (MA)	
Hoybide (UK)		Kennametal (USA)	Yes
Kennametal Hertel (Germany)		Teledyne (USA)	Yes
American National Carbide (USA)	Yes	Duramet (USA)	
CERMeP (France)		Danite (UK)	
BAM (Germany)		Neepsend (UK)	
Marshalls HM (UK)		Japan CCA	
Boart Longyear RC (S Africa)	Yes	CW Carbides (UK)	
Hydra (UK)			

• Feature Article •

GUIDELINE FOR OBTAINING STATISTICAL ADVICE/CONSULTATION

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VAMAS TWA 18 on "Statistical Techniques for Interlaboratory Studies and Related Projects" has as one of its tasks to provide service to VAMAS TWA's. This guideline describes the scope of the service and the procedure for obtaining access to the service.

Scope

Applied mathematics and statistics in the fields of materials science and chemistry. From the point of view of an experimentalist, advice/consultation covers:

- experimental design
- modelling
- simulation
- data analysis

From the point of view of a mathematician/statistician, advice/consultation covers:

- methods of applied and computational statistics, including Bayesian and robust approaches
- methods of computational analysis, including FE-methods and numerical solution of partial differential equations
- software, including the common packages for numerical analysis, statistics/experimental design, computer algebra, and simulation.

Statistical reviewing of testing protocols

Statistical reviewing of testing protocols is recommended in the "VAMAS Project Initiation Form" used by all TWA's when initiating a new work project. All applications for review should be sent to the Chairman of TWA 18 who will identify a TWA 18 member or an external specialist for the review. The TWA Chairman will be kept informed of all correspondence associated with review activities. A formal review request form will not be used because the reviewer will often need specific detailed information on the specific work project in order to determine the adequacy of the proposed statistical methods. Generally, one individual involved in the project will act as liaison with the TWA 18 reviewer.

General problem solving

For a specific question or problem, refer to the list of TWA 18 members (previously distributed to all TWA Chairmen) and their areas of expertise to identify the appropriate expert for the problem. If a match is found, consult the address list and correspond directly with the expert. If a match cannot be found, contact the TWA 18 Chairman for assistance in identifying an external specialist.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 18
"A Review of Accelerated Durability Tests"
by R. P. Brown, RAPRA Technology Limited, Shrewsbury, UK
D. Kockott, Consultant, Hanau, Germany
P. Trubiroha, BAM, Berlin, Germany
W. Ketola, 3M Company, St. Paul, MN, USA
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Edited by R. P. Brown

By definition durability of a material or product implies its performance over a long time scale. The expected life span of polymer products varies considerably but is generally measurable in years and can be 20 or even 50 years. With such time scales it becomes virtually impossible to prove a product under service conditions over its whole design life and it becomes necessary to rely on accelerated tests to predict long term performance.

There is hence an enormous need for reliable accelerated testing methods and prediction techniques which is only matched in magnitude by the inherent difficulties of designing tests which can be relied upon to give meaningful predictions. Whilst large amounts of durability data are produced by accelerated methods, relatively little has been validated as realistically representing service and there is great scepticism on the part of both polymer suppliers and end users as to the value of any accelerated data. Nevertheless there is much valuable information which has resulted from studies of polymer durability which can be applied to help maximize the value of any accelerated testing programme. It is the object of this Guide to make such information readily available.

This Review formed part of the work programme of VAMAS Technical Working Area 12 (TWA12) and followed from a detailed survey that examined the availability, status and usage in the VAMAS countries of accelerated durability tests and is aimed at assisting the development of an ISO standard. It includes information on degradation agents and mechanisms, published standards, designing an accelerated durability testing programme, predictive models, variability and uncertainty, heat ageing, weathering, exposure to liquids and gases, ionizing radiation, pitfalls, and simulated design life exposure of products.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 21
"A Compilation of Introductory and Research Articles"
by K. Doerffel, Martin-Luther-Universität Halle-Wittenberg, Germany
and Thomas Fritz, BAM, Berlin, Germany

VAMAS activities organised through international collaborative projects neccessitate a high level of measuring and analytical procedures as well as powerful techniques for analysis of data. A working group, TWA 18, "Statistical Techniques for Interlaboratory Studies and Related Projects" was founded in 1994 to support this work. The main subjects of this working group are the treatment of theoretical methods and numerical procedures of applied and computational statistics. This includes a service for other VAMAS working groups, optimization of methods and their application, problem-oriented research and consulting in the field of statistics and related subjects. An important task will be the support of discussions between mathematicians, scientists, engineers, and others. Nineteen experienced scientists from 12 countries currently form this group. They are working in various fields of measurement and evaluation covering a major portion of the problem domain.

In this report, research results from some of the scientists in this group are presented in order to introduce the group to the VAMAS community. The report contents are described below:

TABLE OF CONTENTS

I. Interlaboratory Studies

Max Feinberg	*Multivariate Statistical Methods Applied to Interlaboratory Studies*
Susan Steinborn	*Application of Taguchi's Ideas in Interlaboratory Studies*
Klaus Doerffel Heinz W. Zwanziger	*Multivariate Evaluation of an Interlaboratory Test*

II. Experimental Design and Data Analysis

Sonja Arpadjan-Geneva Klaus Doerffel	*Comparison of Second Order Designs for Modelling of Analytical Processes*
Andrzej Parczewski	*Examination of the Interference Effects and Calibration in Chemical Analysis*
Masahito Shimazaki Kazuyuki Suzuki	*Estimability of MLE and Powers of Likelihood Ratio Test on Weibull Regression Model*

III. Calibration

Thomas Svensson	*Uncertainty Considerations in Simplified Transducer Calibration*
Jan Mocak	*Standard and New Procedures for Determining the Limits of Detection and Quantification*

Editors note:

Since the publication of VAMAS Technical Report No 21, it is regretfully reported that Prof Doerffel has died. Until a permanent replacement has been appointed, Thomas Fritz, from BAM in Germany, has taken on the role of acting Chairman of TWA18.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

"Advanced Technical Ceramics:
Trilingual Compilation of Terms - English, French, German"
by J. Lexow, BAM, Berlin, Germany
European Commission, Institute for Advanced Materials
No. CD-NA-16071-EN-C

The trilingual compilation of terms for advanced technical ceramics is the product of a cooperation between CEN Technical Committee 184, Advanced Technical Ceramics, and the VAMAS Technical Working Area 14, The Technical Basis for a Unified Classification System for Advanced Ceramics. The terms were gathered from the literature and various published works. The main sources were a thesis on terminology by Mademoiselle Cecile Plouard, the thesaurus of the data base SILICA by the former Technical Information Center - Materials, Berlin, and the CEC Subcontracting Terminology - Ceramics Sector. The Compilation is held on a data base at the Federal Institute for Materials Research and Testing (BAM) where it has been extended by the inclusion of some definitions from standards and other sources.

Technical Working Area 2

SURFACE CHEMICAL ANALYSIS

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Objectives

- To provide the measurement infrastructure required for setting standard methods of specifying surface chemical analysis
- To develop an agreed base for principles, definitions and equations for relevant aspects of surface analysis techniques
- To identify reference procedures for materials, data, instrumentation and measurement methods

Progress of the technical work in this programme continues to be strong. The overall problem of how to develop and certify reference materials for quantifying dynamic SIMS analyses in Project 5 [details of all TWA 2 Projects can be found in Bulletin No. 19, July 1995] appears to be drawing to a solid conclusion. The method will eventually lead to calibration of the highest accuracy where the resource effort warrants it. The basis of this work is currently in draft for submission to ISO TC201 SC4 for consideration. In many application areas this high level of accuracy is not critical and a new standardless quantification method has been proposed as a new project by Dr. van der Heide from the University of Western Ontario.

This new project is the first to be launched in TWA2 under the new programme structure for VAMAS. If this methodology is proved to the required accuracy, the need for reference materials in a number of areas may be circumvented. This will enhance the value of the technique, lead to the development of instruments, and the greater application of the method in industry.

A second new project covers a major problem area for many analysts. This problem, of surface charge stabilisation and surface charge referencing for insulators, is often held to be trivial in presentations at scientific meetings but, in the author's experience, is still a major and genuine problem in industrial situations. Unfortunately, industrial users cannot always chose to study well-behaved and well-shaped materials and much time may be spent trying to optimise conditions in order to obtain any analysis at all. With some materials, analysis may not be possible. Some instruments may be better than others. This project focuses on

XPS but the problem is equally severe for AES. A solution to this long extant problem will be of major benefit to all industrial users.

A third new project concerns the first topic in the static SIMS area. Static SIMS is a very powerful technique providing considerable detailed information of the bonding and speciation at surfaces, particularly for organics and polymers. However, results are more widely variable than for all other methods. An interlaboratory study was thus proposed in March 1996 to study repeatability, reproducibility and the problems of charge compensation for insulators.

Work progresses in the major project areas of the calibration of XPS energy scales and of defining the composition depth profile in the surface layers by angle-resolved XPS. Here a convergence is developing between Projects 3 and 14. Project 3 provides for the deduction of the composition-depth profile from measurements of the XPS peak intensities at several emission angles. The science leading from a certain depth profile to a set of intensities is not in dispute here but the stability and effectiveness of the inverse calculations are. Four such algorithms are currently being evaluated to provide recommendations for users. In Project 14 the approach uses the inelastic scattering background as a signature of the depth profile. This method has the advantage of not requiring extra measurement time since it can be evaluated from the widescale spectrum. The advantages and disadvantages of these two approaches does need clear evaluation even though both are still in the process of development.

The Standard Data Transfer Format of Project 10 has now been submitted as a draft international standard to ISO TC 201 SC3. Additional to that will be ISO information formats so that data may be properly collated in data banks for later access. For this and Project 30, the Science and Technology Agency of Japan is now developing a client/server Surface Analysis Database. It is likely that in these procedures, commonality in accessing data will be the most important of all of the developments in analysis generally. Also submitted as draft ISO standards is the work for energy calibrations of XPS instruments energy scales, following which will then be a similar document for AES energy scales. These have evolved from Project 2 and will both progress through the joint WG2 for ISO TC201 SC5 and SC7.

Technical Working Area 3

CERAMICS

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Objectives

- To undertake pre-standardization research on the reliability and reproducibility of test procedures for advanced technical ceramics

A cumulative list of all projects undertaken since the inception of this TWA are summarized in Table 1. A final report on Project No. 8, Fracture Toughness of Ceramic Composites has been prepared by the Japan Fine Ceramic Center (JFCC). Almost all of the projects have contributed directly to, or influenced the standards listed in the last column of the table.

Three new projects are underway in 1996.

The National Industrial Research Institute of Nagoya and the Japan Fine Ceramic Center are coordinating a project on conventional hardness of ceramic composites. The objective of the project is to check the applicability of several standardized test methods, prepared for monolithic ceramics, to ceramic composites. Vickers and Knoop indentation methods will be applied to a silicon carbide whisker-reinforced silicon nitride composite. Participants will receive three specimens: silicon nitride only; silicon nitride with 10% whiskers; and silicon nitride with 20% whiskers. Indentations will be made in groups of five, on different surfaces and orientations in the specimens. Vickers indents will be made at 1 and 10 kgf; the Knoop indents, at both 1 and 2 kgf if possible. Altogether, 120-180 indentations will be made by each of twenty laboratories.

A different ceramic hardness project will be coordinated by the Federal Institute for Materials Research and Testing (BAM) in Berlin, in cooperation with the National Institute of Standards and Technology, USA. In the low load testing regime, there is considerable interest in measuring depth of penetration simultaneously with the application of load. BAM has organized a project to study the "Recording (or

Table 1 Cumulative TWA 3 Projects

No.	Title	Organizing Laboratory	Years	Standards
1	Dynamic Fatigue Strength	NIST, USA	1987-1990	
2	Hardness	NPL, UK	1988-1989	CEN ENV 843-4 ASTM C-1326 ASTM C-1327
3	Room Temperature Fracture Toughness	JFCC, Japan	1989-1992	JIS R1607 ASTM C-xxxx[a]
4	High Temperature Fracture Toughness	JFCC, Japan	1990-1993	JIS R1610
5	Quantitative Microscopy	NPL, UK CTK, NL	1991-1992	CEN ENV 623-3
6	Fracture Toughness by SCF Method	NIST, USA	1992-1993	ASTM C-xxxx[a]
7	Fractographic Analysis	NIST, USA EMPA, Switz.	1993-1994	ASTM C-1322
8	Fracture Toughness of Ceramic Composites	JFCC, Japan	1994-1995	
9	Hardness of Ceramic Composites	NIRIN, Japan	1995-	
10	Recording Hardness	BAM, Germany	1995-	
11	Quantitative Microscopy (porosity)	NPL, UK CTK, NL	1996	

[a] Standard under development

instrumented) Hardness of Ceramics". A BK-7 optical glass and a silicon nitride ceramic standard hardness reference block will be used. Participants will be asked to monitor displacement of a Vickers indenter as a prescribed loading rate with maximum forces of either 1N or 10N. Berkovich indenters will be allowed as alternatives. The quantitative microscopy project will be a joint undertaking of the Center for Technical Ceramics, the University of Eindhoven and the National Physical Laboratory, UK. This will be a program co-organized for the VAMAS program and CEN Technical Committee TC 184, Advanced Technical Ceramics. Participants will apply both manual and image analysis methods to characterize the porosity and second phase content of a computer-drawn microstructure, a photograph of a real microstructure, and an actual ceramic specimen.

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[1]
- To provide recommendations for a best practice for routine testing of metallic materials
- To provide pre-cursor information for the development of a LCF reference material

A major BCR/VAMAS project on high temperature low cycle fatigue (LCF) was conducted between 1985 and 1990[2]. The most significant conclusion that emerged from this project was completely unexpected and caused great concern among participants. The concern was that, whilst repeatability of lifetime data within a single laboratory was within a factor of two, reproducibility between laboratories exceeded a factor of sixty. This clearly undermines the reliability of many existing LCF databases and, indeed, the value of any results emanating from a single test system. This is especially so because of the current lack of any independent means of verifying the quality of test results. Subsequent theoretical analysis[3-6] has demonstrated that this poor reproducibility of data can be explained if all the tests had been performed with twice the bending permitted by the ASTM E606 standard. This standard is widely used by industry worldwide and it is not inconceivable that such bending occurred.

The main thrust of the new project is to address this concern. The objectives include (a) the quantification of uncertainties in uniaxial LCF lifetime data due to errors in measuring and controlling the following parameters: (i) misalignment in the machine load-train that causes bending in the test specimen, (ii) strain, and (iii) temperature;

(b) the comparison of theoretical predictions with experimental results; and (c) the provision of recommendations for a best testing practice coupled with a methodology to define a "Statement of Uncertainty" for LCF testing. It is anticipated that this work will be used as a basis for a CEN/ISO standard.

The work started in March 1995 with an EC project entitled *"Quantifying data uncertainties and the validation of a Code of Practice for the measurements of bending in uniaxial fatigue test pieces"*. The project currently involves 25 European industrial and research organizations from 11 countries. The project's work plan is being developed to incorporate work to be performed by additional VAMAS participants. Three Japanese organizations led by NRIM have confirmed their interest to take part and any further participation will be pursued. The EC Main Participants are:

National Physical Laboratory (Project Coordinator), UK
Fachgebiet Werkstoffkunde, T H Darmstadt, DE
Institutet for Metallforskning, SE
BMW Rolls-Royce, DE
TNO Metals Research Institute, NL
Istituto per la Tecnologia dei Materiali, IT
VTT Manufacturing Technology, FI
Inst. Mech. Mat Geostr., GR

References

1. "Code of Practice for the Measurement of Bending in Uniaxial Low Cycle Fatigue Testing", Best Practice in Measurement Series, NPL MMS 001:1995 [ISBN 0 946754 16 0], National Physical Laboratory.

2. Thomas, G B and Varma, R K, "Evaluation of low cycle fatigue test data in the BCR/VAMAS intercomparison programe". Report EUR 14105 EN, BCR Information Applied Metrology, ISSN 1018-5593, Commission of the European Communities, 1993.

3. Kandil, F A and Dyson, B F, "Systematic study of effects of load misalignment in uniaxial low cycle fatigue testing at high temperatures", NPL Report DMM(A)27, July 1991.

4. Kandil, F A and Dyson, B F, "The influence of load misalignment during uniaxial low-cycle fatigue testing I: Modelling", Fatigue Fract Engng Mater Struct, Vol. 16, No. 5, pp. 509-27, 1993.

5. Kandil, F A and Dyson, B F, "The influence of load misalignment during uniaxial low-cycle fatigue testing II: Applications", Fatigue Fract Engng Mater Struct, Vol. 16, No. 5, pp. 529-37, 1993.

Technical Working Area 16

SUPERCONDUCTING MATERIALS

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Objectives

- 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

A special VAMAS session of three technical papers was scheduled at the ICMC Conference in Columbus, Ohio, in July 1995. In conjunction with the ICMC meeting, the 2nd meeting of TWA 16 was held and regional progress reported. In the first Japanese round robin test (RRT) program of high temperature superconducting (HTS) materials, the data exhibited large scatter as a result of an insufficient quantity of material. A second RRT was announced to begin in late 1995. The 1st RRT results from the USA exhibited reasonable results and a second RRT on I_c may not be needed.

In Europe, four RRT's were planned for I_c, T_c, Q_{xyz}, and the NIST simulator. The RRT's on I_c and the NIST simulator had been initiated with 10 participating laboratories. Five laboratories have completed their work. The I_c results on eight HTS samples from four companies exhibited large scatter, however, some samples showed changes of only a few percent after multiple cool downs. Most samples exhibited severe degradation (40 to 60%) before reaching a constant level of I_c. Although the samples were coated with varnish, cracks appeared after many cool downs. A few samples in the RRT have been replaced. The University of Twente has developed a cryogenic "standard" sample that is now part of the RRT. The RRT's on T_c and Q_{xyz} are in preparation and are planned for initiation in late 1995.

The TWA is now in Phase 2 of its program. In Phase 3, a world-wide intercomparison of measurement (ICM) is planned on a limited number of samples based, wherever possible, on a unified set of testing parameters, including sample holder, material, coating, bonding, soldering, cooling rate, etc. A good approach would be to select one stable sample from each continent.

In October 1995, at the request of the Italian Steering Committee delegation, a special

VAMAS meeting was held at ENEA, Frascatti, Italy. The TWA Chairman, Dr. Wada, reviewed the activities of TWA 16. In attendance were representatives from ENEA, Casaccia; IRTEC, Faenza; Institute “G. Colommetti”, CNR, Torino; and ENEA, Frascatti.

The VAMAS report on the I_c measurement intercomparisons was published as a Supplement in Volume 35 of “Cryogenics”. The title is “VAMAS, The Versailles Project on Advanced Materials and Standards, Technical Working Area No. 6, Superconducting and Cryogenic Structural Materials Report: Vol. 1, Critical Current Measurement Method for Nb_3Sn Multifilamentary Composite Superconductors”.

The next meeting of the TWA will be in 1996 at the Applied Superconductivity Conference in the USA.

Technical Working Area 17

CRYOGENIC STRUCTURAL MATERIALS

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Objectives

- Exchange of information on mechanical testing methods of composite materials at cryogenic temperatures
- Intercomparison of compression and shear properties of graphite-fiber reinforced plastic at cryogenic temperatures and establishment of reliable testing methods
- Intercomparison of tensile and fracture toughness properties of an aluminum alloy at cryogenic temperatures and establishment of reliable testing methods
- Establishment of recommended testing methods at cryogenic temperatures

The 10th meeting of the cryogenic structural materials working party was held on 17 July, 1995 in Columbus, Ohio, USA. Dr. Ogata summarized the round robin test (RRT) programs for both the composite material (G-10CR) and the aluminum alloy (2219). Nine laboratories from five countries are participating in the composite testing program and eight laboratories from five countries are participating in the aluminum alloy program.

Aluminum Alloy

The average yield strength and ultimate tensile strength strength were 472 MPa with a standard deviation of 9 MPa (1.9%) and 666 MPa with a standard deviation of 9 MPa (1.3%), respectively. These results show that this aluminum alloy exhibits lower strength and toughness compared to previous results with a titanium alloy and 316LN stainless steel and prove that there are few problems in cryogenic tensile tests for these materials.

Composite Material

Preliminary compression test results were reported. The average compressive strength and Young's modulus were 763 MPa with a standard deviation of 119 MPa (16%) and 18 GPa with a standard deviation of 2.6 GPa (14%), respectively. The average shear strength was 129 MPa with a standard deviation of 19 MPa (15%). There appears to be little difference between results from the short beam shear test and the guillotine shear test. Discussion focused on the effect of buckling and failure mode and on material available, specimen preparation, testing conditions, and reports for the second RRT.

Future Schedule

Composite material (2nd RRT)
Material: The amount of G-10CR available was reported as follows:

Thickness (mm)	Number of sheets
25.6	3.5
12.7	3.5
6.3	2
2.5	10

Sheet dimensions: 610 mm x 455 mm

The 6.3 mm sheet remains in short supply and efforts are being made to obtain more material for distribution among the participating laboratories.

Dimensions of Specimen: The definition of specimen ratio, length/diameter or length/area were discussed, and with the analysis of the first RRT results, the length/area criterion was selected for the 2nd RRT and it must fall within the range of 0.1 to 0.3 based on the ASTM slenderness ratio definition. For the short beam shear test, the span ratio should be 4.

Specimen machining: Specimens will be machined (to participants dimensions), packed in silica-gel to control humidity and distributed by NRIM. Participants can also machine their own specimens from a bulk sample if desired.

Testing temperature: Participants agreed to carry out the 2nd RRT at liquid nitrogen temperature (77K) and, if possible, also at liquid helium temperature (4K).

Test Reports: Reports should include a description of the failure mode in both compression and shear tests together with the ultimate compression strength, failure strain, Young's modulus, and Poison's ratio.

Alloys

Participants had carried out a series of RRTs to refine the testing procedure according to the strength-toughness matrix: high strength and higher toughness; high strength and lower toughness; and lower strength and lower toughness. Solder is a soft material, with very low strength and unknown toughness at low temperature. Participants can check and calibrate the accuracy of their testing system in the low loading strain region with solder. A detailed discussion of the purpose and procedure led most participants to agree to a RRT and to initiate tensile tests on solder. The material will be prepared by FZK in Germany and machined by NRIM. The chosen alloy is commonly used for soldering superconducting wire. Tests will be carried out at 4K.

Other activities

The final draft report on the strain gage program has been prepared by IMGC and it will be submitted to Cryogenics for publication. The following TWA meeting was planned for the occasion of the ICEC/ICMC 96 meeting at Kitakyushu, Japan in May 1996.

VAMAS Calendar

TWA 17 (Cryogenic Structural Materials) Meeting in conjunction with the ICEC/ICMC 96 Meeting, Kitakyushu, Japan	May 1996
TWA 21 (Mechanical Test Methods for Hardmetals) in conjunction with Advances in Hard Materials Production meeting, Stockholm, Sweden	May 1996
ISO TC 201 Meeting, near Guildford, Surrey, UK	July 11-13, 1996
2nd International Meeting of Pacific Rim Ceramic Societies, PacRim 2, including meeting of ISO TC 206 (Fine Ceramics), Cairns, Australia	July 1996
QSA9 (Surface Chemical Analysis), Guilford, Surrey, UK	July 15-19, 1996
TWA 20 (Measurement of Residual Stress), AECL, Chalk River, Canada	September 1996
43rd AVS Meeting (Surface Chemical Analysis) immediately following ASTM E42 Meeting, Philadelphia, PA, USA	October 14-18, 1996
7th European Conference on Applications of Surface and Interface Analysis, ECASIA 97, Göteborg, Sweden	June 16-20, 1997

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