VAMAS

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## November 2000

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

Website: www.vamas.org

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: Atomic Force Microscope (AFM) image of the thermally faceted surface of polycrystalline aluminum oxide. The large flat facet in the center is the basal plane as evidenced by the hexagonal shape. The image is approximately 5 micrometers across and the height is 200 nanometers.

*Photograph courtesy of John Blendell, Ceramics Division,*
*National Institute of Standards and Technology*
*Technology Administration*
*U.S. Department of Commerce*

VAMAS

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# • Chairman's Message •

It is a pleasure to be able to report to you as the current Chairman of the VAMAS Steering Committee. The Secretariat passed from NPL to NIST in 1999, with myself as Chair and Jim Early as Secretary. I must thank and congratulate Kamal Hossain the previous Chairman for his numerous efforts in strengthening VAMAS and moving the organization in new directions. During his tenure as Chairman, Kamal began a process of developing a Strategic Plan for VAMAS that would guide us over the next few years. The Mission and Goals of the Strategic Plan have now been agreed to by the Steering Committee. They are as follows:

MISSION
To support world trade in products dependent on advanced materials technologies by providing the technical basis for harmonized measurements, testing, specifications, and standards.

GOALS
- Optimize the selection of VAMAS topics based on standardization needs.
- Enhance recognition of VAMAS.
- Increase impact of VAMAS.

The plan contains more detailed objectives and tasks which VAMAS can undertake in order to achieve these above goals. Our success will depend on a 'team' effort by both TWA participants and the Steering Committee.

VAMAS is also moving into several new technical areas through the formation of new TWAs. These include TWA 24 on "Performance Related Properties for Electroceramics", TWA 25 on "Creep/Fatigue Crack Growth in Components", TWA 26 on "Full Field Optical Stress and Strain Measurement Methods", TWA 27 on "Characterization Methods for Ceramic Powders and Green Bodies", and TWA 28 on "Quantitative Mass Spectroscopy of Synthetic Polymers" (pending final approval). Remember to visit the VAMAS web site at www.vamas.org for updates on new activities.

I believe that VAMAS continues to be a strong and effective organization, in large part due to the dedication and hard work of the TWA Chairs and all of the individuals involved in the research activities. Jim and I look forward to working closely with you over the next couple of years.

Steve Freiman
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.

Recent adopted standards include:

1] ISO 14606, ***Sputter Depth Profiling-Optimization using Layered Systems as Reference Materials,*** 2000, ISO Technical Committee 201, Surface Chemical Analysis.
VAMAS contributor - TWA 2, Surface Chemical Analysis, C. Powell, Chairman.

2] ISO FDIS 15472, ***X-ray Photoelectron Spectroscopy-Calibration of Energy Scales,*** 2000, ISO Technical Committee 201, Surface Chemical Analysis.
VAMAS Contributor - TWA 2, Surface Chemical Analysis, C. Powell, Chairman.

3] ISO FDIS 14975, ***Surface Chemical Analysis-Information Formats,*** 2000, ISO Technical Committee 201, Surface Chemical Analysis.
VAMAS Contributor - TWA 2, Surface Chemical Analysis, C. Powell, Chairman.

4] ASTM C1421-99, ***Standard Test Methods for the Determination of Fracture Toughness of Advanced Ceramics,*** 1999, ASTM Committee C 28, Advanced Ceramics.
VAMAS Contributor - TWA 3, Ceramics, G. Quinn, Chairman.

5] ISO 14705, ***Test Method for Hardness of Monolithic Ceramics at Room Temperature,*** 2000, ISO Technical Committee 206, Fine Ceramics.
VAMAS Contributor - TWA 3, Ceramics, G. Quinn, Chairman.

6] JIS R 1617, ***Testing Method for Fracture Toughness of Fine Ceramics at Elevated Temperature,*** 1999, Japanese Industrial Standard Committee.
VAMAS Contributor – TWA 3, Ceramics, G. Quinn, Chairman

# • Feature Article •

## NEW TECHNICAL WORKING AREA

## TWA 24 Performance Related Properties for Electroceramics

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### INTRODUCTION

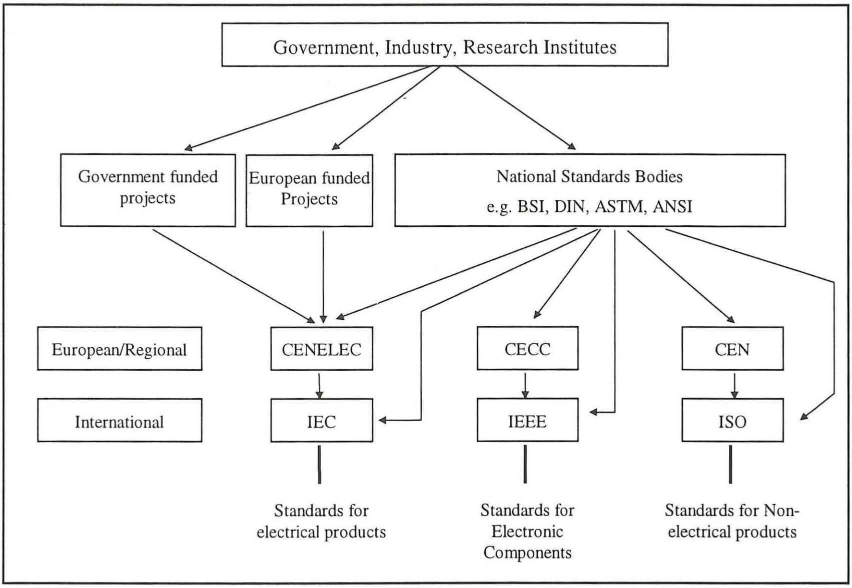
Electroceramics such as piezoelectric and electrostrictive materials have the capability of converting electrical energy into mechanical energy (or vice versa). The technological importance of these materials is increasing, with widespread use in actuator and sensor applications. Although there has been some standardisation activity through bodies such as the IEEE and more recently through CENELEC in the development of new standards, many of the most important properties that are required for these materials when used as sensors and actuators remain without internationally recognised test methods. Measurements where work is required include direct and converse piezoelectric coefficient measurement, high stress dielectric property measurement, the measurement of strain at high stresses, and the measurement of degradation of materials performance under repeated electrical and mechanical loading.

### CHALLENGES

Piezoelectrics operate in more demanding environments near limits of their linear behaviour. Finite Element (FE) Analysis is needed to explore their properties and performance under these conditions. Materials property data at such stresses, fields, frequencies etc are not generally measured. Software techniques are coming on line that can describe the properties as complex coefficients and this may help describe their non-linear performance. The hysteretic properties also hinders use and knowledge of these losses and will aid designers in developing better, more reliable products. Thus the challenges include: tool kit of measurement good practices for piezoelectric and other functional materials embracing high field non-linear property evaluation; FE models and implementation that can use these data in designing new materials and products; training in these areas and education.

## STANDARDS ALREADY AVAILABLE

It is instructive to describe how standards organizations interact with each other on a national and international scale. The following schematic describes how several nationally based and European based standards committees interact with International bodies, that are relevant to materials based research. Other countries have similar systems in place.




There are a number of international standards written for piezoelectric material measurements including the established IEEE 176 that is used most frequently. Additionally, US military and other standards exist for some of the more specialized applications such as sonar systems employing piezoelectric ceramics. A list of the standards that are more frequently used are listed below:

- IEEE Standard on Piezoelectricity IEEE Std 176-1978.
- IRE Standards on Piezoelectric Crystals, 1961, Proc. IRE, pp. 1162-1169; July 1961.
- IEC standard publication 483, 1976. 'Guide to dynamic measurements of piezoelectric ceramics with high electromechanical coupling'.

STANDARDS UNDER DEVELOPMENT

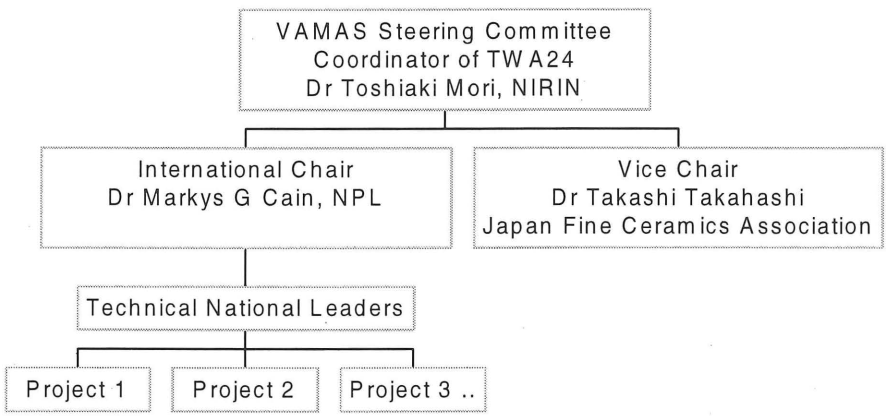
- Pre-normative CENELEC Standard 63-2, 'Piezoelectric Properties of Ceramic Materials and Components: Part 1 and Part 2', describes measurement methods for low and high field dielectric properties. CENELEC, BTTF 63-2, developed by BTTF, Advanced Technical Ceramics working group, has produced 3 documents for bulk materials and is now working on multilayer components. The scope of future work includes thick films.

- IEEE Loss in Acoustic Materials subcommittee: Details at http://www.ieee-uffc.org/about/uffc_std_policy.html

## REMIT FOR THIS TWA

The remit for this new TWA are those areas that are not currently covered by existing standards or where existing standards are out of date and need expanding. This TWA aims to explore various areas with the view to submit recommendations to appropriate standards bodies: IEEE, CEN, and BSI. The structure of this TWA is shown in the schematic below.

**VAMAS TWA 24 - Electroceramics**




## HISTORY AND STATUS OF VAMAS 24

The concept of a new TWA in electroceramics pre-standardisation was taken in January 1999. An e-mail based questionnaire was written and sent to major electroceramic users and producers around the world. There was overwhelming support for the setting up of this TWA. The TWA was approved at the 1999 VAMAS Steering Committee meeting.

The projects that were supported from the e-mail questionnaire were further clarified and projects described in enough detail to establish the first project. Project No.1 is the Measurement of the Piezoelectric Coefficient of Materials. International support and participation in this project would be based on the initial supporting organisations and by the National Leaders. The Japanese National Leader, Dr Takahashi, will manage Project No. 1. The National Leaders listed at the end of this article will orchestrate their country's involvement in projects.

The global awareness and impact of VAMAS has been raised at a recent US Office of Naval Research Review (April, 2000 at Penn State University). Much interest was demonstrated and potential new project ideas and participation identified. This type of activity will form a main dissemination route for TWA 24.

## PROPOSED PROJECTS

Initially it is expected that efforts will focus of development of procedures for the following project:

| | |
|---|---|
| Project 1 | Measurement of piezoelectric coefficient of piezoelectric materials of differing geometries and configurations, including assessment of direct and converse coefficients |

The following projects and conceptual ideas have been proposed and are at an early planning/discussion phase.

| | |
|---|---|
| Project 2 | Measurement of piezoelectric strain at high electrical/mechanical stress |
| Project 3 | Measurement of piezoelectric and dielectric properties at high stress |
| Project 4 | Measurement of electrical and mechanical fatigue of piezoelectric ceramics materials |
| Project 5 | Properties of electrically conductive, optical transparent thin films |
| Project 6 | Thermal effects on performance |
| Project 7 | The dielectric, elastic and piezoelectric properties (matrix elements) measured as complex coefficients so as to take account of the electrical, mechanical and piezoelectric losses in the material. The complex coefficients should and can be measured as a function of frequency. |
| Project 8 | The temperature dependence of the complex coefficients is important in some applications (such as high power and space applications). |
| Project 9 | Hydrostatic property measurements to about 14 MPa are important for underwater applications. |
| Project 10 | The meaning of fatigue / aging / degradation need clarification, particularly the distinction between reversible and irreversible mechanical or electrical damage |
| Project 11 | In project 1, introduce measurements of piezoelectric coefficient of thin and thick films on substrates. |

## INTERACTIONS WITH OTHER TECHNICAL WORKING AREAS

Close liaison will be maintained between TWA 3 and TWA 24 because participating organisations may have dual interests both in electronic and structural ceramic materials.

The VAMAS TWA 24 web is accessible from the following home page and includes a single page summary of TWA 24 as well as a general description of VAMAS: http://www.vamas.org

The TWA 24 home pages are located at the Functional Materials Group Website at NPL. This web site is: http://www.npl.co.uk/npl/cmmt/functional/VAMAS.html

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**HOW TO BECOME INVOLVED**

The TWA is working to enlist the co-operation of measurement laboratories that are interested in evaluating methods for determining performance-related properties of electroceramics. Please e-mail me your research interests and participation areas. Include your organization's structure and whether you would be willing to manage a project.

# • Feature Article •

## NEW TECHNICAL WORKING AREA

## TWA 25 Creep/Fatigue Crack Growth in Components

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### INTRODUCTION

VAMAS has been active in the field of standardisation of fracture mechanics testing and analysis at elevated temperatures since 1987. Between 1987-1992 a new working group TWA 11, Creep Crack Growth, was setup to develop and formulate a standard for a high temperature test method. Recommendations were made for measuring the creep crack growth properties of materials and using the creep fracture mechanics parameter $C^*$ in the analysis of the data. The method was restricted to ductile creep situations. The findings were incorporated into ASTM test procedure E1457-92, the first standard to deal with crack growth testing at elevated temperatures.

This methodology was extended under TWA 19, High Temperature Fracture of Brittle Materials (1993-1998) to conditions where only limited creep deformation or otherwise creep brittle conditions were observed. As a consequence of a round robin testing and analysis programme on four relatively creep brittle alloys, recommendations were made to modify the original procedure and incorporate the methodology for more creep brittle circumstances. Subsequently a revised version of the ASTM standard E1457-98 was produced. Furthermore E1457-98 is about to be replaced with a new revised version which will take into account nearly all VAMAS TWA 19 recommendations. It will cover the wider range of creep ductile to creep brittle testing conditions observed in engineering alloys.

As a result of experience gained from TWA 11 and TWA 19, the present TWA 25 was established in June 1999 with the broad aim of recommending testing, analysis and life prediction methods for assessing elevated temperature creep and creep/fatigue crack growth in metallic components containing defects. Professor T Yokobori, Teikyo University, Japan, agreed to serve as Co-Chairman.

The overall objectives of TWA 25 are defined as follows:

- Establish accurate and reliable procedures for determining creep and creep/fatigue crack growth at elevated temperatures in components which contain defects.
- Determine best procedures for analysing the test data using fracture mechanics concepts.
- Provide validation of results against measurements on standard laboratory specimens using the ASTM E1457-98.
- Propose relevant models for life assessment methods for cracked components.

## INDUSTRIAL BACKGROUND

Manufacturer's recommendations and their past experience have usually been the basis for the design of vital engine components such as turbine blades, vanes and discs and in critical engineering components such as gas steam pipes, pressure vessels and in weldments which might contain pre-existing defects. In recent times however crack growth initiation and failure analyses have become more acceptable as an independent design and remaining life assessment methodology. The development of high temperature fracture mechanics concepts, through which the time dependent effects of creep could be modelled, uses experimental uniaxial and crack growth data from simple laboratory tests specimens in order to predict failure times under operating conditions. Furthermore the improvement in non-destructive inspections and testing methods (NDT) has allowed smaller and smaller defects to be detected and the need for more reliable methods for predicting crack initiation/incubation periods and steady crack growth rates.

The final objective of developing testing procedures is to improve the reliability of life assessment codes that use test information. In developing a testing standard methodology for laboratory specimens (ASTM E1457) a first step was taken to improve life prediction procedures of components. However life extension calculations of components requires a validated fracture mechanics model for crack initiation and growth as well as detailed knowledge of component non-linear time dependent stress analysis, past service records and postulated future operations together with 'appropriate' mechanical properties. It therefore seems appropriate to develop a testing method for components and integrate it with life assessment codes for creep and creep/fatigue of components.

## BACKGROUND TO LIFE ASSESSMENT CODES

Components in the power generation and petrochemical industry operating at high temperatures are almost invariably submitted to static and/or combined cycle loading. They may fail by net section rupture, crack growth or a combination of both. The development of codes in different countries has moved in similar directions and in many cases the methodology has been borrowed from a previously available code in another country. The early approaches to high temperature life assessment show methodologies that were based on defect-free assessment codes. For example ASME Code Case N-47 and the French RCC-MR which have many similarities are based on lifetime assessment of un-cracked structures. More recent methods make life assessments based on the presence of defects in the component. For example the German flat bottom hole approach (FBH) considers crack detection and characterisation and the German two-criteria method regards initiation as an important factor in life assessment and does not deal with the crack growth regime. The more advanced codes dealing with defects over the range of creep and creep/fatigue interaction in initiation and growth of defects are the British R5, BSPD-6539 recently replaced by BS 7910 and French A16 which have clear similarities in terms of methodology.

Figure 1 shows a schematic diagram of the range of crack initiation and growth behaviour that a cracked component might show. Generally it can be divided into two regions. Firstly the initiation region whose limit can be determined either from micro-mechanical models or from NDT limits and secondly the steady crack growth region which can be described using the fracture mechanics parameters such as *K*, reference stress $\sigma_{ref}$ and $C^*$. The more recent defect assessment procedures mentioned above are based on experimental and analytical models to assess crack initiation and growth and to determine the remaining useful life of such components. These codes base their analysis on tests taken from laboratory specimens that are invariably derived from small specimens at short test times. Therefore there is no direct verification of the predicted results with component testing. This is an important point since size and geometry differences impose various degrees of constraint that affects crack growth and initiation.

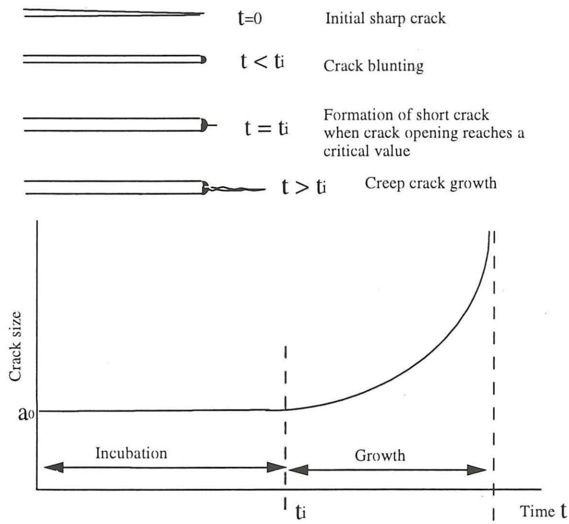


Figure 1: Schematic behaviour of a crack at elevated temperature

In addition it is clear from these assessment methods that the correct evaluation of the relevant fracture mechanics parameters, for which the lifetime prediction times are dependent upon, are extremely important. It is also evident that the detailed calculation steps, which are proposed in these documents, do not in themselves improve the accuracy of the life prediction results. In any event as these procedures have been validated for limited sets of geometries and material data their use in other operating conditions will need careful judgement.

The two British high temperature codes BSPD-6539 (now replaced with the more recent BS 7910) and British Energy's R5 attempt to deal comprehensively with assessment and remaining life estimation procedures that can be used at the design stage and for in service situations. They focus on a life assessment approach allowing the expert to decide upon the applicability of the predictions in relation to the operating circumstances. The concept implies that the codes need to show they are both reliable and understandable over a range of material and loading conditions that may not have been previously examined or validated by the code developer. This is particularly important as new higher strength steels, which have little or no long term material properties data base, are developed or used by the power industry.

Therefore the trend in the development of the codes suggests that, in addition to verification of data between laboratory tests and component tests, increased flexibility in dealing with the information and the analysis is an important factor. This acknowledges the fact that calculations however detailed and sophisticated will not necessarily come up with the correct predictions due to various unknowns in assessment procedure. These can be attributed to a number of factors that may be beyond the control of the engineer using the code. They are as follows

- The available material property data for the analysis are invariably insufficient or crude and since they are usually taken from either historical data, results from different batches of material or tested in different laboratories with insufficient number of tests specimens they are likely to contain a large scatter.
- The scatter and sensitivity in creep properties inherently produce a large variation in the calculations. Upper and lower bounds are therefore introduced which give widely different life prediction results.
- The evaluation of the relevant parameters such as $K$, limit load concepts, reference stress $\sigma_{ref}$ and $C^*$ are different according to the method of derivation.
- The use of short-term small laboratory data for use in long term component life predictions further increases the possibilities of a wrong prediction.
- Difficulty in ascertaining the level of crack tip constraint and multi-axiality effects in the component will reduce the accuracy of crack growth predictions by about a factor of 30.
- Unknowns in modelling the actual loading history, component system stresses and additional unknowns such as little or no knowledge of past service history, residual stresses also act as sources of error in predictions.
- Non-destructive (NDT) methods of measuring defects in components, during operation and/or shutdown and insufficient crack measurement data during operation, is likely to add to errors involved in life-time assessment.

All these factors suggest that however detailed, sophisticated and accurate a particular calculation is, the result will still need to be treated with caution. In addition the similarity of the approaches in the various codes do not necessarily imply that calculations by the different methods will give the same predictions. It may be possible that under certain controlled and validated circumstance the predictions can be optimised. It is clear that a critical comparison is only possible when the same method is used on another material and condition or the same test cases are examined by the different codes.

TWA 25 will attempt to fill this gap in order that modelling methods and test data from standard laboratory and feature component tests can be used with increased confidence in life estimation codes. Early indications are that both the relevant ASTM and ASME bodied have shown interest in the progress of this project. Clearly the recommendations resulting from this project will be useful for both of these institutions.

**OBJECTIVES FOR TWA 25**

On the basis of the established background of creep and creep/fatigue crack growth test methods and also life assessment methodology that has been discussed a programme of work has been setup in TWA 25.

The main objective is to establish accurate and reliable procedures for assessing creep crack growth at elevated temperatures in components that contain defects. Determine procedures for analysing the test data using fracture mechanics concepts. Validation of results against measurements on standard laboratory specimens using the ASTM E1457-98. Finally, in the light of established results, to propose recommendations to both testing methods for components and changes to life assessment codes.

Since TWA 25 was started, two international meetings have been held. The first was in London in June 1999 and the second in Tokyo in November 1999. It has been clear that there is substantial interest shown by the power generation industry, in particular, in developing this field. Participants from Europe, Japan and USA have attended the two meetings and a number of others who have not been able to attend have requested to be placed on the e-mail list. The following institutions are the core group constituting the committee:

| | | | |
|---|---|---|---|
| Teikyo University | Japan | EPRI | USA |
| Tohuku University | Japan | GKSS | Germany |
| NRIM | Japan | SPG, Dresden | Germany |
| IHI | Japan | CEA | France |
| MHI | Japan | JRC Petten | Holland |
| Hitachi | Japan | British Energy | UK |
| Toshiba | Japan | ERA Technology | UK |
| Nippon Steel Corp. | Japan | Bristol University | UK |
| JSW | Japan | ABB-Alsthom | UK |
| Wright/Patterson AFB | USA | Mitsui-Babcock | UK |
| Georgia Tech | USA | ETD | UK |
| MPC/PURC | USA | Imperial College | UK |

The overall programme for TWA 25 is spread over 4 years. The description and the plan for implementing the objectives are described below;

## PRE-STANDARDISATION NEEDS

For design and safety assessments, industry needs assurances that creep crack growth properties measured on laboratory specimens can be applied to real components. Laboratory specimens usually contain through thickness cracks whereas components mostly experience partially penetrating defects. The size of a component and the stress state it is in may also be different from that of the laboratory specimens. Some experiments have already been carried out on 'feature' components which represent realistic situations, to measure crack growth but no agreed procedure exists for interpreting the results. The aim therefore is to address this issue by performing analysis of these results. No tests are planned on components because of expense.

## PROJECT PLAN

The plan of the project will be

a) gather together experts from industry and research institutes in order to identify their specific needs with respect to feature component testing.
b) produce a data-base of available feature component test data and on-going tests.
c) Identify acceptable feature components and best practice for undertaking tests.
d) Establish reliable methods for the analysis and interpretation of the data.
e) Develop methods of calibrating the results in terms of material crack growth properties data of standard fracture mechanics specimens.

## CONCLUSIONS

**The background to the present TWA 25 has been presented and it has been established that there is a need in industry to improve life assessment methods in terms of creep and creep/fatigue crack initiation and growth in components which operate at elevated temperatures. Therefore a programme of works has been established where the emphasis has been initially placed on collecting information and experience from participating partners. The collection and the development of this data-base will dictate, to a great extent, the decisions regarding the next round of this project. Early indications are that there is firm industrial support for TWA 25 and it is hoped that over the next three years positive collaboration from members will make this a successful TWA.**

# • Feature Article •

## NEW TECHNICAL WORKING AREA

## TWA 26 Full Field Optical Stress and Strain Measurement

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### 1. INTRODUCTION

A new TWA has been established to bring together the experimental stress analysis community and develop standards for the use of stress and strain measurement techniques. The methods which are to be considered are:

- Photoelasticity
- Moiré methods
- Laser speckle and interferometry methods
- Image correlation
- Thermoelastic methods

Integration with Finite Element (FE) methods for the purpose of validating numerical analyses will form an additional part of the work of this TWA.

### 2. INDUSTRIAL BACKGROUND

The past ten to twenty years has seen an enormous expansion in the development and availability of full field stress and strain measurement and analysis methods, which have potentially a very high value to industry. The current requirements to satisfy standards and regulatory authorities mean that engineers must test and validate models of structures with greater rigour. To do this, engineers need to use methods that have been suitably validated. In addition increasingly difficult problems in design and development of new structures arise from a number of factors including reduced cost and development time.

Full field optically based stress analysis methods in conjunction with numerical analysis must be seen as the safest, fastest and most cost effective way of solving these problems. The use of these methods also leads to a deeper understanding of the engineering problems and a reduction in the quantity of expensive testing. Enormous confidence can be gained from the correlation between measured stress and strain data and numerical analyses over the full field of a component. The adoption of this philosophy is essential in developing engineering knowledge as it provides a more certain basis for the design of engineering components. It is also superior to the current situation, where cross correlation between techniques is only carried out at discreet points on the structure.

The vast majority of these full field techniques are optically based interferometric methods, which give direct or indirect measures of the surface deformations, stresses or strains. The main reason for the recent rapid development of these methods is the advent of smaller, cheaper and more powerful image collection and processing equipment, the most important of which is arguably the personal computer. Evidence of this immense interest is given in the recent publication by the IMechE devoted almost entirely to this class of techniques [1]. This journal edition contained seven papers of which six deal with full field optical techniques. The total number of references in these six papers was 374 and this list is by no means exhaustive.

In addition to the significant amount of time devoted to research in these areas there is also great interest in industrial applications of these techniques. In particular, the development of an automated reflection polariscope [2] has been supported strongly by the aerospace industry as have ESPI (Electronic Speckle Pattern Interferometry) and shearography [3]. Shearography has already been introduced to the industrial production process for quality control of helicopter rotor blades [3].

The challenge, which the stress and strain analysis community must address, can be met by unification of the community to develop a cogent set of standards. These standards will form a framework within which industry can apply these methods and develop them further. It is important to obtain recognition and acceptance of full field optical techniques so that further investment in their future development can come from those who will specifically benefit. This investment will only come if industry can use the methods in a way that is accepted by the standards and regulatory authorities.

## 3. CURRENT STANDARDS

At present no national or international standards exist which cover the procedures, materials and equipment used for any of the full field optical techniques. Some acceptance of these techniques exists in the Aerospace industry for certification purposes, but there is no overall acceptance of any of the four or five different classes of technique.

One of the many benefits of developing international standards for these methods would be their subsequent acceptance by the certification or regulatory authorities. The importance of this cannot be over emphasised as it is becoming more critical that structural, process and material analysis are performed more cheaply, in a shorter time and to a higher degree of certainty than ever before. The increasing complexity of these problems also means that numerical analysis methods cannot be relied upon exclusively, the need for adequate validation using physical measurement methods is therefore becoming more important.

An example of how some standardisation has helped in the proliferation and acceptance of strain measurement methods is given by Measurements Group [4]. Measurements Group provide a substantial library of information on strain gauge application, coating technology, data collection and processing routines. In addition, the code of practice [5] published by the British Society of Strain Measurement is an attempt to formalise procedures as well as the equipment used which is covered by a variety of standards. These two examples could be used as a blueprint of what is required for the automated full-field stress and strain measurement methods. A significant advance on this sort of data library would be to have it validated and disseminated by an appropriate standards authority.

## 4. AREAS OF COMMON INTEREST

Full field optical strain measurement techniques share a great deal of features and procedures. This is partly due to their basis in interferometry of some kind, which leads to the production of periodic maps of quantities such as deformation or strain difference. In addition, the equipment used to collect and process the image data is similar and so the associated problems are shared to some degree.

The outline below is a first attempt to describe some of the areas into which standards in optical techniques could be rationalised. In many cases the working methods and equipment specifications have already been defined and accepted in the stress analysis community. It is, however, important that a complete and thorough classification is performed and that these are published in a format that is more widely accessible.

The purpose of the following lists is to show the large and varied number of aspects that need to be considered.

***Equipment***

- Light sources
  - Spectral distribution
  - Monochromatic/polychromatic
  - Lasers
- Cameras/Image sensors
  - Detector type (CCD, CID, CMOS)
  - Pixel shape
  - Pixel response characteristics
- Lenses, mirrors and other critical and non-critical generic optical components
- Digitiser cards
  - Resolution (spatial and spectral)
  - Digitisation noise
  - Data compression

***Reference materials***

- Physical reference materials
  - Polarisers and quarter wave plates
  - Gratings
  - Coatings
- Calibration specimens
- Virtual reference materials
  - Simulated data
  - Synthesised fringe patterns
  - Noisy Data Synthesis

*Output standards*

- Image data format
- Numerical data format
- Processed data format(stress, strain, fracture mechanics parameters)

*Operational procedures*

- Specimen preparation
- Application of coatings
- Loading; range, strain rate etc
- Ambient conditions(e.g. temperature, light)
- Alignment of specimens

The behaviour and characteristics of the equipment associated with the measurement is crucial to the accuracy and repeatability of the results (as are procedures). The sort of equipment currently being used has a well-defined specification. The effect, on the data collected, of variations within these specifications is not clearly known and understood. With the development of standards it would not be necessary to change or limit the specifications but to understand and describe more clearly their effect on the data produced.

The importance of reference materials is that it allows the equipment and techniques to be calibrated against a known standard so that measurements taken on different equipment can be reliably compared. This not only forms the mainstay of traceability which is very important in aircraft certification but would also help in comparing new methods as they are developed. It would also lead to a quantitative description of how good a particular system or component is with reference to another system or previous version.

Although it is widely accepted that all stress and strain analyses cannot be performed to a strictly prescriptive set of procedures, a set of standards would indeed be beneficial. In the case of traditional techniques, such as strain gauging and photostress coating, the procedures set out by Measurements Group [4] are generally considered to be the standard. This does not mean that these are adhered to in all cases, but when they are a significant mark of authority is achieved.

## 5. PRE-NORMATIVE RESEARCH

In addition to VAMAS there has been a great deal of interest in developing measurement and testing standards through the BCR (Bureau Communitaire de Reglements) of the European Commission (EC) [6]. Many projects have been supported which have led in part to the development of standards [7]. Support from the EC for the sort of project being proposed here is likely to be forthcoming under the new Fifth Framework programme.

The Commission is supporting this type of activity as is made clear in the publication "Research and Standardisation" [8] that was produced recently. This document outlines why standards are becoming increasingly necessary and where the EC wants to focus research activity to develop standards. In addition it wants to enhance the effectiveness of this research to maximise the transfer of pre-normative research into actual standards. The new TWA expects to take advantage of this favourable environment to promote and support its work.

The following table shows a provisional list of participants.

| Institution | Country | Type | Methods |
|---|---|---|---|
| National Research Council | CANADA | U/R | All |
| SNECMA | FRANCE | U | PE |
| LMSGC | FRANCE | R | PE/MM |
| Ettemeyer GmbH & Co | GERMANY | E/(U)/R | LS |
| DaimlerChrysler AG | GERMANY | U | PE/MM |
| MTS Corporation | USA | E | |
| Univ. of Pisa | ITALY | R | PE/TE/IC |
| Univ. Aoyama Gakara | JAPAN | R | PE |
| Univ. Wakayama | JAPAN | R | MM |
| BAe | UK | U/R | PE/MM/LS/IC |
| Univ. of Sheffield | UK | E/R | PE/TE/MM |
| Univ. of Strathclyde | UK | R | MM/PE/TE |
| IVECO | ITALY | U | All? |
| Boeing | USA | U | PE/TE |
| Measurements Group | USA | E/U | PE/MM |
| Univ. of Jaen | SPAIN | U/R | PE/MM/LS |
| Honlet Optical Systems | LUXEMBORG | | All? |
| Univ. of North Carolina | USA | R | |
| National TsingHua Univ. | Taiwan | R | |
| National Physical Laboratory | UK | U/R | MM/LS/FE |

Key
U—User R—Researcher E—Equipment Manufacturer

| | | |
|---|---|---|
| LS | — | Laser speckle methods, e.g. ESPI, Shearography |
| PE | — | Photoelasticity (reflection, transmission etc) |
| MM | — | Moiré methods (shadow, projection, geometric, interferometric, etc) |
| IC | — | Image/speckle correlation |
| TE | — | Thermoelastic (SPATE, DeltaTherm) |
| FE | — | Finite Element Analysis (integration, CAE etc) |

## 6. CONCLUSIONS

A number of optically based stress and strain measurement methods are developing rapidly and being introduced into industry. Greater use of these techniques and their continuing use could be enhanced by the development of standards. This would give clear signals that the industry is developing in a structured manner. With the support currently being offered by agencies such as the European Commission, it is the right time enter into pre-normative research. It is also beneficial to exploit the fact that the stress analysis community is still relatively small and has not suffered fragmentation due to divergent interests. It is therefore prudent to act now while these conditions prevail. It will also be beneficial to avoid problems in the future such as have been experienced in the optics industry [9] because of the lack of standards.

A suitable set of norms will mean that the methods referred to above will have a level of traceability and repeatability essential when dealing with certification and conformance issues such as those governed by regulatory authorities. In the aerospace industry, for example, a set of standards would significantly facilitate the acceptance of full field optical stress and strain measurement methods by the certification authorities. The widespread use of such methods in aerospace as well as other industries will be beneficial for users and system manufacturers alike. In the longer term this will help to improve the impact of the stress analysis community if it has the backing of internationally accepted standards.

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# • Feature Article •

## NEW TECHNICAL WORKING AREA
## TWA 27 Characteisation Methods for Ceramic Powders And Green Bodies

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### 1. INTRODUCTION

A new Technical Working Area (TWA) has been established with the broad aim of developing and standardizing methods for the characterization of ceramic powders and green bodies because they are important for ceramic manufacturing processes. In this regard, the proposed work encompasses three different areas:

- methods for powder properties
- methods for suspension characterisation
- methods for green body characterisation

Activities in these areas are important for enhancement of future successful industrial development by improving international trade in ceramic products. The path to be followed includes the standardization of test methods to characterize the different physical properties.

### 2. INDUSTRIAL BACKGROUND AND NEED

To date only a few methods for characterisation of ceramic powders and porous materials have been standardised. This concerns all materials, but is especially valid for advanced ceramics. The present situation is related to the complexity of the subject due to the influence of many parameters on powder properties during measurements. These include chemical impurity and inhomogeneity of the powders, and geometrical features of particles and agglomerates (shape, size and size distribution). In order to make further advances in the development of new materials, improvements are needed in characterisation methods for different stages of the manufacturing processes. This is particularly true for characterisation of powders during preparation and processing of raw materials and for intermediary stages of manufacturing where powders exist in suspension, like slurries, as well as during the shaping and forming process used for the preparation of green bodies.

## 3. STANDARDISATION EFFORTS

Standardisation committees for advanced ceramics exist in various regional, national and international standardisation bodies, including ASTM C 28, JIS R, JIS Z, DIN NMP 291, CEN TC 184, ISO TC 206 and ISO TC 24. Cooperation exists between these committees, and links to the proposed VAMAS activity have already been established through the participation of members of different committees.

### 3.1 Current situation of national standards related to ceramic powders

Japan :
Japan has started an initiative to promote the technical infrastructure of the fine ceramics industry [1]. Included is the concept of the Open Material System (OMS) which aims develop a better understanding of manufacturing processes including processing, as well as design guides and databases. The first stage of this program is projected to end in 2001, with the second stage starting at the same time. Table 1 gives an overview over the currently established Japanese industrial standards (JIS) related to ceramic powders.

Table 1: Overview of the Japanese Industrial Standards (JIS) relevant to TWA 27.

| | | |
|---|---|---|
| JIS | 1619 | Testing method for size distribution of fine ceramic particles by liquid photo-sedimentation method |
| JIS | 1620 | Testing methods for particle density of fine ceramic powder |
| JIS | 1622 | General rules for the sample preparation of particle size analysis of fine ceramic raw powder |
| JIS | 1626 | Measuring methods for the specific surface area of fine ceramic powders by gas adsorption using the BET method |
| JIS | 1629 | Determination of particle size distributions for fine ceramic raw powders by laser diffraction method |
| JIS | 1633 | Sample preparation method of fine ceramics and fine ceramic powders for scanning electron microscope observation |
| JIS | 1638 | Test methods of iso-electric point of fine ceramic powders |
| JIS | 1639-1 | Test methods of properties of fine ceramic granules; Part 1 : Size distribution of granules |
| JIS | 1639-2 | Test methods of properties of fine ceramic granules; Part 2 : Bulk density |
| JIS | 1639-3 | Test methods of properties of fine ceramic granules; Part 3 : Drying loss |
| JIS | 1639-4 | Test methods of properties of fine ceramic granules; Part 4 : Flowability |

Europe:
The situation in the European Union with regard to standardisation of advanced ceramics is characterised by the work of European standardisation committee CEN TC 184 during the past decade. Standardisation efforts are found in five established working groups:

WG 1: Classification
WG 2: Ceramic Powders
WG 3: Monolithic Ceramics
SC 1: Long-Fibre Composites
WG 5: Ceramic Coatings

Accordingly the work of WG 2 is related to the work of the new TWA 27. Table 2 lists the standards developed and published so far that relate to powders.

Table 2: Overview of standards and drafts related to VAMAS TWA 27.

| | | |
|---|---|---|
| EN | 725 | Methods of test for ceramic powders |
| EN | 725-1 | Determination of impurities in alumina powders |
| ENV | 725-2 | Determination of impurities in barium titanate powders |
| EN | 725-3 | Oxygen content of non-oxide powders |
| ENV | 725-4 | Determination of oxygen content in aluminium nitride powders by XRF analysis |
| EN | 725-5 | Particle size distribution |
| EN | 725-6 | Determination of the specific surface area |
| EN | 725-7 | Determination of the absolute density |
| EN | 725-8 | Determination of tapped bulk density |
| EN | 725-9 | Determination of untapped bulk density |
| EN | 725-10 | Determination of compaction properties |
| ENV | 725-11 | Determination of the densification on natural sintering |
| prEN | 725-12 | Determination of impurities in Zirconia |
| prENV | 725-13 | Determination of crystalline phases in Zirconia |
| prENV | 725-14 | Determination of flowability behaviour of ceramic granule |
| WI | 92 | Determination of impurities in silicon nitride, Part I |
| WI | 129 | Determination of impurities in silicon nitride, Part II |

United States:
In the United States numerous technical committees of ASTM have been instrumental in developing standards for characterisation of powders and particulate materials. The techniques covered by these standards span a broad range of technologies and are used by various industry segments, including ceramics, powder metals, paper and pulp, chemical manufacturing, pharmaceutical, environmental, and petroleum. Various standards pertaining to particle characterisation cover the following areas [2]:

- Representation and Treatment of Data
- Sedimentation, Classification, Gravity and Centrifugal Methods
- Surface Area and Porosity Measurement Methods
- Sieving Methods
- Electrical Sensing Methods
- Laser Diffraction Methods
- Image Analysis Methods

- Single Particle Light Interaction Methods
- Sampling Methods
- General Methods and Information

Table 3: List of ASTM standards related to the scope of TWA 27.

| | | |
|---|---|---|
| ASTM | C721 | Test Method for Average Particle Size of Alumina and Silica Powders by Air Permeability. |
| ASTM | C958 | Test Method for Particle Size Distribution of Alumina or Quartz by X-Ray Monitoring of Gravity Sedimentation. |
| ASTM | C1282 | Test Method for Determining the Particle Size Distribution of Advanced Ceramics by Centrifugal Photo-sedimentation. |
| ASTM | C1274 | Test Method for Advanced Ceramic Specific Surface Area by Physical Adsorption. |
| ASTM | C1096 | Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption. |
| ASTM | D1993 | Test Method for Precipitated Silica-Surface Area by Multi-point BET Nitrogen Adsorption. |
| ASTM | D4284 | Standard Test Method for Determining Pore Volume Distribution of Catalysts by Mercury Intrusion Porosimetry. |
| ASTM | C371 | Standard Test Method for Wire-Cloth Sieve Analysis of Nonplastic Ceramic Powders. |
| ASTM | C925-79 e1 | Standard Test Method for Precision Electroformed Wet Sieve Analysis of Nonplastic Ceramic Powders. |
| ASTM | C690 | Standard Test Method for Particle Size Distribution of Alumina or Quartz by Electric Sensing Zone Technique. |
| ASTM | F661 | Standard Practice for Particle Count and Size Distribution Measurement in Batch Samples for Filter Evaluation Using an Optical Particle Counter. |
| ASTM | F662 | Standard Test Method for Measurement of Particle Count and Size Distribution in Batch Samples for Filter Evaluation Using an Electrical Resistance Particle Counter. |
| ASTM | F795 | Standard Practice for Determining the Performance of a Filter Medium Employing a Single-Pass, Constant-Rate, Liquid Test. |
| ASTM | F660 | Standard Practice for Comparing Particle Size in the Use of Alternative Types of Particle Counters. |

## 3.2 Activities of other related international organisations or committees

### ISO TC 206, Fine Ceramics

This committee has been working on standardisation of advanced ceramic materials on an international level since 1992. Its work covers all areas of advanced ceramics[3]. Table 4 lists the standards under development and preliminary work items (PWI) related to powders.

Table 4: Standard drafts and projects of ISO TC 206.

| | | |
|---|---|---|
| CD | 18757 | Determination of specific surface area of ceramic powders by gas adsorption using the BET method |
| CD | 18753 | Determination of absolute density of ceramic powders by pycnometer |
| PWI | 2 | Determination of particle size distribution of ceramic powders by laser diffraction method |

**ISO TC 24 /SC 4, Sizing by Methods Other than Sieving**

This international committee works on standardisation of sizing methods independent of the material system. Table 5 lists those standards that have already reached the inquiry stage or are established.

Table 5: Standards related to VAMAS TWA 27 under development by ISO TC 24.

| | | |
|---|---|---|
| DIS | 9276-2 | Representation of results of particle size analysis - calculation of average particle sizes/diameters and moments from particle size distributions |
| DIS | 9276-4 | Representation of results of particle size analysis; Part 4: Characterization of a classification process used for particle size analysis |
| DIS 2 | 13317-1 | Determination of particle size distribution by gravitational liquid sedimentation methods; Part 1: General principles and guidelines |
| DIS | 13317-2 | Determination of particle size distribution by gravitational liquid sedimentation methods; Part 2: Fixed Pipette methods |
| DIS | 13317-3 | Determination of particle size distribution by gravitational liquid sedimentation methods; Part 3: x-ray method |
| DIS | 13318-1 | Determination of particle size distribution by centrifugal sedimentation methods; Part 1: General principles and guidelines |
| DIS | 13322 | Particle size analysis - Image analysis methods |
| DIS | 13323-1 | Determination of particle size distribution Single particle light interaction methods; Part 1: Light interaction considerations |
| FDIS | 14887 | Particle size analysis; Sample preparation - Dispersing procedures for powders in liquids |
| ISO | 9276-1 | Representation of results of particle size analysis; Part 1: Graphical representation |
| ISO | 9277 | Determination of the specific surface area of solids by gas adsorption using the BET method |
| ISO | 13319 | Particle size analysis - Electrical sensing zone method |
| ISO | 13320-1 | Particle size analysis - Laser diffraction methods; Part 1: General principles |
| ISO | 13321 | Particle size analysis - Photon correlation spectroscopy; Part 1: General principles and guidelines |

## 4. FUTURE PLANS

The above description shows that standardisation activities relating to ceramic powders have made significant progress during the last ten years. Areas not yet covered relate mainly to the direct processing and manufacturing steps with regard to the granules, slurries and green bodies. In this area standardisation has been difficult mainly for two reasons:

- area of sensitive know-how
- area of technical difficulty because methods are not developed enough for standardisation and further research is necessary.

Most methods for the characterisation of nanosized powders belong to this latter area. The main task of TWA 27 will be to establish international cooperation to enhance the reliability of those methods which are the basis for expanding standardisation into the areas of "secondary powder properties" and green bodies. Items of interest in this regard are:

Rheological Properties of Slurries
Dispersion State of Slurries
Moisture Content of Granules
Strength of Green Bodies
Density Gradients in Green Bodies
Porosity and Pore Size Distributions
Machining Damage in Green Ceramics
Binder Distribution
Density Measurements of Complex Shapes

## 5. PARTICIPANTS

Interest in this topic is indicated by the participation of the following institutional and industrial partners during the past ten years under an International Energy Agency (IEA) program, and could be potential participants in TWA 27.

United States:
National Institute of Standards and Technology (NIST)
Sandia National Laboratories
Alfred University
Micromeritics, Inc.
Advanced Cerametrics
Superior Technical Ceramics.

Belgium:
Vlaamse Instelling voor Technologisch Onderzoek (VITO).

Germany:
Bundesanstalt für Materialforschung und –prüfung (BAM)
University Clausthal
Keramische Werke Hermsdorf
Robert Bosch GmbH
Max-Planck Institute PML
Fraunhofer Institute ISC.

Sweden:
Swedish Ceramic Institute
Ytkemiska Institutet (YKI)
Permascand.

| | |
|---|---|
| Japan: | Nagaoka University of Technology (NUT)<br>National Industrial Research Institute of Nagoya (NIRIN)<br>Osaka National Research Institute (ORIN)<br>Nihon Tokushu Togyo Kabushikikaisha (NTK)<br>Nihon Gaishi Kabushikikaisha (NGK)<br>Kyocera, Asahi Glass Company<br>Japan Fine Ceramics Center (JFCC). |
| Yugoslavia: | Institute of Technical Sciences, Serbian Academy of Sciences and Arts. |
| Switzerland: | Eidgenössische Materialprüfungs- und Forschungsanstalt (EMPA). |

It is hoped that this new TWA will also attract interest from other countries, who are hereby invited for participation.

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# • Recent VAMAS Outputs •

## EXTENDED ABSTRACT

VAMAS Technical Report No. 37
"Fracture Toughness of Ceramics using the SEVNB Method;
Round Robin"
by J. Kübler, EMPA, Swiss Federal Laboratories for Materials
Testing and Research, Dübendorf, Switzerland

[A joint round robin of VAMAS TWA 3 and
European Structural Integrity Society (ESIS TC6]

Fracture toughness was measured by the Single-Edge-V-Notched Beam (SEVNB) method on five monolithic advanced technical ceramics in an international round robin. These ceramics were coarse- and fine-grained alumina (alumina-998, alumina-999), gas pressure sintered silicon nitride (GPSSN), sintered silicon carbide (SSiC) and yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), and had different degrees of difficulty in the application of this test method. Very consistent results were obtained for the alumina-998. The fracture toughness for the 135 tests accepted as valid tests from 28 participants was 3.57 ± 0.22 MPa $m^{1/2}$. Reasonably consistent results were obtained for the alumina-999. The fracture toughness for the 102 tests accepted from 21 participants was 3.74 ± 0.40 MPa $m^{1/2}$. Consistent results were obtained for the GPSSN. The fracture toughness for the 129 tests from 27 participants was 5.36 ± 0.34 MPa $m^{1/2}$. Very consistent results were obtained for the SSiC. The fracture toughness for the 56 tests accepted from 12 participants was 2.61 ± 0.18 MPa $m^{1/2}$. As predicted, less consistent results were obtained for the Y-YZP due to its grain size in the submicron range. The fracture toughness for the 35 tests accepted from 7 participants was 2.61 ± 0.18 MPa $m^{1/2}$.

Only the mean for the alumina-998 differed significantly from other credible test methods. A combination of a high sensitivity to subcritical, slow or stable crack growth near the V-notch tip and a pop-in of small cracks to form a crack "initiation" seem to be reasonable for the discrepancy.

The SEVNB method proved to be forgiving and robust with respect to the notch preparation, notch width (<10 μm), notch depth or optical quality for ceramics with and average grain size or major microstructural feature size of greater than about 1 μm. Most participants had no difficulties and rated the method user-friendly, reliable and worthwhile for standardization.

# • Recent VAMAS Outputs •

## EXTENDED ABSTRACT

VAMAS Technical Report No. 38
"Neutron Diffraction Measurements of Residual Stress
in a Shrink-fit Ring and Plug"
by G. Webster, Imperial College, London, UK
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R. Lin Peng, Uppsala University, Nyköping, Sweden

Edited by G. A. Webster

This project was initiated to develop a recommended procedure for making residual stress measurements in crystalline materials by neutron diffraction. It involves a series of round robin investigations to establish the resolution and reliability of the technique. This report presents the findings on the first set of measurements which were carried out on a shrink-fit aluminum alloy ring and plug assemble. The assembly was made by first cooling an oversize plug to allow it to be fitted into a ring and then allowed to return to room temperature to generate residual stresses in the ring and plug. A ring and plug assembly was chosen as the first sample to be measured because the residual stresses can be introduced elastically, a discontinuity is obtained at the ring/plug interface and comparisons can be made with theory.

Although neutron diffraction measurements have been made at 18 neutron sources, the measurements at each source were made in isolation. Most of the measurements were made on steady-state instruments that used a monochromatic beam of neutrons. The remainder were made on time-of-flight instruments which used a pulsed monochromatic beam. Comparable results were obtained from each type of instrument. With a monochromatic source measurements are made on specific crystallographic planes; with the time-of-flight method the entire spectrum can be analysed using profile refinement to obtain strains.

Initially a protocol was produced for making the measurements. Precautions were specified to ensure accurate positioning and alignment of a sample in the neutron beam. A suitable shape and size of 'gauge volume' over which individual measurements were to be made was defined to achieve adequate resolution in regions of strain gradients. A recommended procedure for recording results was also specified to assist in establishing reliability.

A detailed statistical study of the results has been performed using Bayesian analysis to obtain 'best estimates' of the measured strains and therefore calculated stresses. With this analysis, it has been established, in most cases, that a positional accuracy with a standard deviation of 0.1 mm can be achieved. It has also been ascertained that strains can be recorded away from a surface to an accuracy of better than $10^{-4}$ corresponding to a stress of about 7 MPa in aluminum. Greater errors are obtained close to a surface due to surface aberrations. It has been found that some errors can be attributed to imprecise determinations of stress-free lattice spacings. Corrections to these can be made to satisfy force balance where this is possible. The size of the gauge volume determines the extent to which discontinuities in stress can be resolved. Comparisons with theory suggest that the maximum hoop stress predicted in the ring at the interface of 80 MPa was underestimated by 10 MPa in this investigation due to averaging over the gauge volume. However, extrapolation of the data would virtually eliminate this discrepancy.

The feasibility of making reliable estimates of residual stress by neutron diffraction has been demonstrated and the basis of a recommended measurement procedure established. The procedure will be refined in further round-robin studies being carried out on a ceramic matrix composite, a shot-peened plate and a ferritic steel weldment with a view to producing a draft standard. Although attention has been focussed on the determination of residual stress, neutron diffraction can be used to measure applied stress as well.

# • Recent VAMAS Outputs •

## EXTENDED ABSTRACT

VAMAS Technical Report No. 39
"VAMAS Round Robin on Flexural Strength of Silicon Nitride at High Temperature"
by A. Okada and M. Mizuno, JFCC, Nagoya, Japan

Thirteen laboratories in six countries participated in the VAMAS round robin to measure the flexural strength of silicon nitride at high temperatures. At each laboratory, the four-point flexural strength of silicon nitride at 1200 °C in air was measured in semi- and fully-articulated configurations, depending on laboratories. All together, seventeen different sets of measurements were obtained. The analyses were made for the effect of fixture configurations and for the different combinations of spans between 30 mm x 10 mm and 40 mm x 20 mm.

A comparison between semi- and fully-articulated fixtures having 40 mm x 20 mm spans indicates that a fully-articulated configuration yields higher strengths. Strengths from fully-articulated fixtures averaged 654 ± 101 MPa while strengths from semi-articulated fixtures averaged 622 ± 89 MPa. Although the fully-articulated fixture produces more uniform loading, particularly for poorly machined specimens, twist error predictions do not explain the strength difference. The effect of spans was examined in a semi-articulated configuration. The strength with 30 mm x 10 mm spans averaged 661 ± 75 MPa while that for 40 mm x 20 mm spans averaged 622 ± 89 MPa. The difference is consistent with the effective volume/surface area prediction.

In addition, flexural strength was also measured in nitrogen atmosphere to examine an atmosphere effect. It was found that the flexural strength at 1200 °C in nitrogen is considerable lower than that in air. It was suggested that the flexural strength of silicon nitride at elevated temperature in air is improved by the oxidative crack healing. The round robin results indicated that the results were not affected by variables such as soaking time, furnace configuration, or detailed fixture configuration.

## WEAR TEST METHODS

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At present there are three active projects underway. The objective of Project No. 1, Compilation of Wear Test Standards, is to provide a validated source for standardized wear test methods for use by engineers and tribologists. A database of over 400 standards has been established. The database is being reviewed by VAMAS participants to evaluate the usefulness and robustness of the standards. Obsolete standards will be removed. Comments on the applicability and usefulness of specific standards to industry will be added to the final report.

The goal of Project No. 2, Ball Cratering Wear Testing, is to establish the ball cratering wear test method as a standard and to develop a recommended practice for conducting the test. This abrasive wear testing method is particularly useful for testing coatings. A draft procedure has been written and potential participants have been identified. The project leader has been appointed ASTM task leader for the development of this test into a standard enhancing the transitioning of VAMAS pre-standardization results to a standards development organization (SDO).

The focus of the final project, Project No. 3, Wear Debris Characterization Methods and Representation, is to develop wear debris characterization methods for size, shape, and morphology and to develop a mathematical representation of the debris. Procedures for wear debris generation for round robin tests have been defined and a survey of methods for wear debris analysis and representation prepared by the project leader. The preparation of wear test samples is underway. This is a joint project with TWA 7, Biomaterials.

Possible new work areas include: development of a certified reference material for friction and wear; and calibration of friction measurements on the nano-scale. This latter idea would involve using an atomic force microscope in the AFM mode to measure the height of a surface and in the LFM mode to measure friction on a low scale to reveal more surface structure detail. However, a method of force calibration will be needed.

# SURFACE CHEMICAL ANALYSIS

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Since its inception, TWA 2 has initiated 40 TWA projects. Twenty-one projects have been completed and there are three inactive projects that could not be completed for lack of resources. The 16 current active TWA projects are listed below.

| No. | Project Title |
|---|---|
| 2.* | Development of calibration data for the energy scales of Auger-electron spectrometers |
| 3. | Procedures for quantitative X-ray photoelectron spectroscopy |
| 5.* | Development of reference materials prepared by ion implantation |
| 9.* | Intercomparison of Auger-electron energy and intensity measurements |
| 13. | Tests of algorithms for data processing in AES - Factor analysis and intensity |
| 14.* | (b) Tests of algorithms for background subtraction in AES |
| | (c) Tests of algorithms for quantitative XPS by peak and peak-background shape analysis |
| | (d) Tests of algorithms for angle-resolved XPS |
| 21. | Tests of algorithms for the analysis of multicomponent spectra in XPS |
| 23.* | Absolute calibration of XPS instrument intensity scales |
| 30. | Development of a Common Data Processing System for AES and XPS |
| A1. | Use of the infinite velocity method for SIMS quantification |
| A2. | The evaluation of static charge stabilisation and determination methods in XPS on non-conducting samples |
| A3. | Interlaboratory study of static SIMS repeatability and reproducibility |
| A4. | Evaluation of multilayer reference coatings for quantitative GDOES depth profiling |
| A5. | Interlaboratory study of the degradation of organic materials in XPS analysis |

*These projects received support from the Community Bureau of Reference, CEC.

The states leading active TWA 2 projects are as follows:

| | | | |
|---|---|---|---|
| Canada | A1 | UK | 2, 9, 21, 23, A3 |
| Germany | 5, 13, A2, A4 | USA | 2, 3 |
| Japan | 30, A5 | EC | 14(b), 14(c), 14(d) |

Figure 1 gives a pictorial indication of progress for current projects.

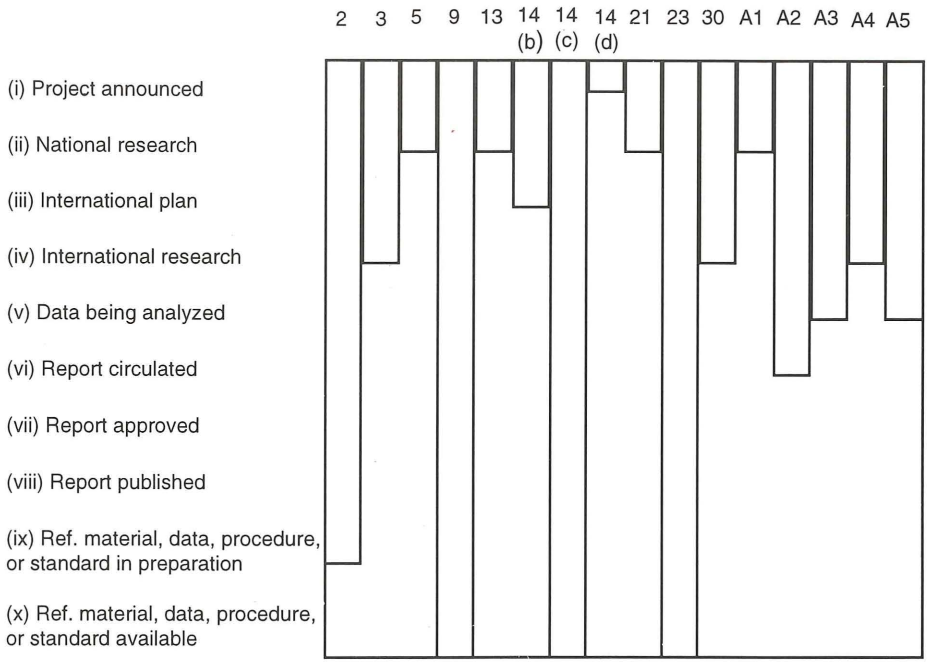


Fig. 1. Diagram showing the progress of TWA 2 active projects.

Many of the outputs of TWA 2 projects have been incorporated or are in the process of being incorporated into ISO standards. Table 2 identifies the standards or draft standards of ISO Technical Committee 201, Surface Chemical Analysis, arising from or related to TWA 2 projects. TWA 2 is a category-A liaison body with ISO/TC 201 and its subcommittees, and there is excellent communication between these groups.

There has been satisfactory progress in the active TWA 2 projects during the past year. Four interlaboratory comparisons are being conducted (projects A2, A3, A4, and A5) for which there are 26, 18, 36, and 38 participants, respectively.

Projects 9, 14(c), and 23 have been completed during the past year. The completion of projects 9 and 23 through the provision of a service by the UK National Physical Laboratory to calibrate the intensity scales of AES and XPS instruments is a major accomplishment.

Table 1. Standards or draft standards arising from or related to TWA 2 projects.

| ISO/TC 201 Subcommittee | Title of standard or draft standard |
|---|---|
| SC2: General Procedures | NP 16268: Ion-implanted surface-analytical reference materials |
| SC3: Data Management and Treatment | ISO 14976: Surface chemical analysis (data transfer)<br>FDIS 14975: Surface chemical analysis (information format)<br>WD 15760: Data dictionary for the description of X- ray photoelectron and Auger electron spectroscopy data records |
| SC4: Depth Profiling | ISO 14606: Surface chemical analysis—Sputter depth profiling (optimization)<br>CD 15969: Surface chemical analysis—Depth profiling (sputtered depth) |
| SC5: AES | NP 18118: Surface chemical analysis—Auger electron spectroscopy and X-ray photoelectron spectroscopy (sensitivity factors) |
| SC7: XPS | FDIS 15472: Surface chemical analysis—X-ray photoelectron spectroscopy (energy scale calibration)<br>CD 17973: Surface chemical analysis—Medium resolution Auger electron spectrometers (energy scale calibration)<br>CD 17974: Surface chemical analysis—High resolution Auger electron spectrometers (energy scale calibration)<br>WD 18327: Surface chemical analysis—X-ray photoelectron spectroscopy (unintended degradation) |

Interlaboratory comparisons conducted some 20 years ago showed that the ratios of measured intensities of different peaks for the same specimen could vary by up to one or two orders of magnitude, for XPS and AES, respectively, on different instruments. The new NPL calibration service should have a significant impact on the quality of future surface analyses by AES and XPS.

The completion of project 14(c) is also a major accomplishment. Another necessary requirement for a reliable XPS analysis is that the photoelectron intensity be measured and analyzed correctly. For many years, scientists have used simple, empirical methods to measure peak intensities (although it was known that these methods did not have physical justification). A physical model has been developed and applied for the transport of the signal electrons in XPS and it is now possible to obtain critical morphological information for the specimen and to measure the compositions of different phases; software for this purpose is now available.

## CERAMICS FOR STRUCTURAL APPLICATIONS

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The round robin on Fracture Toughness by the SEVNB method was completed and a comprehensive final report published. A round robin on Elevated Temperature Flexural Strength testing is underway and proceeding satisfactorily. A new round robin on Determination of Phase Composition and Percent Crystallinity in Hydroxyapatite is at an advanced state of planning. A general overview article: "VAMAS at Twelve" was published by the American Ceramic Society Bulletin in July, 1999. It summarized the 12 round robins and 12,000 experiments that have been completed in VAMAS TWA 3 in the last 12 years.

The list of standards that have been impacted by TWA 3 projects is growing. A cumulative list of all projects undertaken since the inception of this TWA are summarized in Table 1. Projects have contributed directly to, or influenced the standards listed in the last column of the table. This information is also now available on the TWA 3 web site that is accessible from the NIST Ceramic Division home page. The address is: www.ceramics.nist.gov/webbook/vamas/vamastwa3.htm

The site includes a single page summary of the first 12 TWA #3 projects as well as general descriptions of TWA 3 and VAMAS.

Status of Current Projects

1. The Fracture Toughness by the SEVNB method round robin was completed and a final report prepared by the Swiss Federal Laboratories for Materials Testing and Research, Zurich. The method will be included in the new European Standards Committee TC 184 standard test method for determination of fracture toughness. The simplicity of the precracking procedure (a common industrial razor blade with diamond paste) appealed to many users particularly in the industrial sector. This project was a joint VAMAS/European Structural Integrity Society (ESIS) project with 35 participating laboratories.

Table I. Cumulative TWA 3 Projects

| No. | Title | Organizing Laboratory | Years | Standards |
|---|---|---|---|---|
| 1 | Dynamic Fatigue Strength | NIST, USA | 1987-1990 | |
| 2 | Hardness HV, HK, R45N | NPL, UK | 1988-1989 | CEN ENV 843-4<br>ASTM C-1326<br>ASTM C-1327<br>ISO 14705 |
| 3 | Fracture Toughness I, Room Temperature SEPB, IS, IF | JFCC, Japan | 1989-1992 | JIS R1607<br>ASTM C-1421<br>CEN ENV xxxx[A]<br>ISO 15732 |
| 4 | Fracture Toughness II, High Temperature CN, SEPB | JFCC, Japan | 1990-1993 | JIS R1610 |
| 5 | Quantitative Microscopy I Grain Size, Porosity | NPL, UK<br>CTK, NL | 1991-1992 | CEN ENV 623-3 |
| 6 | Fracture Toughness III, SCF | NIST, USA<br>EMPA, Switz. | 1992-1993 | ASTM C-1421<br>CEN ENV xxxx[A]<br>ISO 18766 |
| 7 | Fractographic Analysis, Fracture Origins | NIST, USA<br>ARL, USA | 1993-1994 | ASTM C-1322<br>CEN ENV xxxx[A] |
| 8 | Fracture Toughness IV, Ceramic Composites, SENB, SEPB, SEVNB | JFCC, Japan | 1994-1995 | |
| 9 | Hardness of Composites, HV, HK | NIRIN, Japan<br>JFCC, Japan | 1995-1996 | |
| 10 | Recording Hardness | BAM, Germany | 1996-1998 | |
| 11 | Quantitative Microscopy II, Volume Fraction, Porosity | JFCC, Japan | 1996-1998 | CEN ENV xxxx[A] |
| 12 | Fracture Toughness V, SEVNB | EMPA, Switz. | 1997-1998 | CEN ENV xxxx[A] |
| 13 | Elevated Temperature Flexural Strength | JFCC, Japan | 1999- | ISO 15765 |
| 14 | Phase Composition & Crystallinity in Hydroxyapatite | JFCC, Japan | 1999- | |

[A] Standard under development

2. The High Temperature Flexure Strength project began in October 1999. Thirteen laboratories are participating. This project was organized by the Japan Fine Ceramic Center and is intended to support the development of an elevated temperature flexure strength test standard by ISO TC 206, Fine Ceramics.

3. A new project Determination of Phase Composition and Percent Crystallinity in Hydroxyapatite has been initiated under the leadership of the National Physical Laboratory in cooperation with Queen Mary College, UK. Hydroxyapatite (HA) is a ceramic material increasingly used as a biocompatible monolithic or coating encouraging adaptation of an

implant into the human body. However the body's response depends critically on the phase composition of the material. This round robin will assess the level of accuracy and repeatability achievable by a method proposed for the quantification of HA crystallinity and phase composition. ISO/TC150/SC1 has drafted two international standards for hydroxyapatite ceramics and hydroxyapatite coatings. The VAMAS output will provide an assessment of the reliability of the method proposed for the ISO standards as part of a statement of confidence in the technique.

Proposed Future Projects

The following projects have been proposed and are at an early planning/discussion phase.

1. Surface Roughness
Dr. R. Morrell of the National Physical Laboratory reported that the Phase I, European part of this project was still underway and would need to be analyzed before a joint VAMAS/CEN Phase II project could begin.
*Status of Project*: On hold pending analysis of Phase I project.

2. Thermal Shock
Dr. R. Wäsche of BAM, Berlin proposed that TWA 3 start a thermal shock project. BAM is constructing a laser shock apparatus for thermal shock evaluation of ceramics. It is understood that few other laboratories could have such equipment, but Dr. Wäsche has proposed that laboratories could compare different methods on a common material.
*Status of Project*: Discussion phase. Project to be developed and defined.

3. Nondestructive Characterization
Dr. O. Toft Sorenson of RISOE, Denmark proposed a new project for nondestructive characterization (NDC) of ceramics. The objective is to determine whether micro-defects in ceramics can be characterized effectively by current nondestructive characterization techniques and whether the data obtained by these techniques can be used reliably to predict mechanical strength.
*Status of Project*: Discussion phase. Project to be defined and developed.

4. Elastic Modulus
Dr. R. Morrell of NPL suggested a project for elastic modulus determination. The project would include: beam resonance by impulse excitation, beam resonance by forced excitation, beam static flexure, and ultrasonic time of flight methods. Preliminary work has been conducted in a 5 laboratory EC/CERANORM project. Several key technical issues remain unresolved with these methods.
*Status of Project*: Discussion phase. Project to be developed and defined.

5. Wear resistance
Dr. J-P. Erauw and Dr. Philippe Descamps of the Belgian Ceramic Research Center (CRIBC) have proposed a new project for wear resistance evaluation. CRIBC has a new model for correlation of wear resistance to basic mechanical properties such as elastic modulus, hardness, and fracture toughness. The model contains different hardness and fracture toughness dependencies and includes elastic modulus. The specific test method is open for the time being. The project should be coordinated with VAMAS TWA #1, Wear Test Methods.
*Status of Project*: Discussion phase. Project to be developed and defined.

*Technical Working Area 5*

## POLYMER COMPOSITES

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There are presently three projects underway. A fourth project, Assessment of Damage Tolerance for Polymer Matrix Composites, is being re-defined due to a change in project leadership.

Project 1, Assessment and Recommendations to ISO on Mode II Test Methods, has been completed. The project was organised to assist ISO TC61/SC13/WG16 in overcoming a veto on Mode I progression without Mode II. Based on the Mode II round robin it was recommended and agreed at the September 1999 ISO TC61 meeting that "4-point ENF" procedure is the preferred method for standardisation. Further input from the VAMAS round robin into ISO 13003 is being made as it progresses through standardisation.

Project 2, Measurement of Mechanical Properties for the Fibre/Matrix Interface, seeks to refine micro-mechanics test methods for assessing the fibre-matrix interface strength in composites, beginning with the best known method, single fibre fragmentation. There are 3 objectives: (1) establish an accepted test protocol, (2) conduct a round robin to demonstrate that the protocol enables multiple laboratories to get equivalent results, and (3) generate an extensive database of information on one or more model systems in order to explore different data analysis models.

There is no current standard for interface strength testing although there are a variety of test methods that have been used to determine this parameter. The existing methods have been most successful for relative measurements and consequently, are most useful for quality control and materials/surface treatment selection. Although the tests are used most widely by fibre companies and the aerospace industry, lack of standardization is a major barrier to use by many other companies. There are two critical needs. First, it must be demonstrated that a standardized test procedure can give reproducible results in different laboratories. Second, the analysis procedure to calculate interface strength from the measured results must be improved to deal with realistic materials.

The work in this program addresses the first need directly and develops an extensive database of information on model systems as a first step toward addressing the second need. Two model systems will be used: one ideally suited for the round robin and a second amenable to the Raman studies. Preliminary tests are underway in a single laboratory to validate the test protocol and material. Next the round robin on the first model system will begin with a target date for completion of the program for the first model system at the end of 2000. Round robin tests with the second model system would begin in 2001 with completion of the program with the single fibre fragmentation tests and both model systems scheduled for the end of 2001.

The third project, Measurement of Through-thickness Testing Properties has been delayed due to a change in posts by the project leader. The development of composite materials for structural applications in new sectors such as the ground transportation industry requires the use of thicker structures made of cost-effective materials (thick glass-epoxy woven composites, for instance). However, knowledge of through-thickness properties becomes necessary in order to design such structures and only few poorly understood methods are available. Therefore, there is an important need from industry to have such through-thickness test methods available. NPL has submitted some new drafts to BSI for ISO standards and ASTM has some related items. The associated EU Framework 5 network proposal was submitted early in 2000.

*Technical Working Area 7*

## BIOMATERIALS

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Hard tissue materials such as those of artificial joints are one of the most succesful and widely used material in the medical field. Total joint replacements in human bodies are increasing in frequency world-wide due to increasing life expectancy, population growth, advances in medical sciences, and increasing number of accidents. There are, however, still serious problems in using these materials under the severe mechanical and physiological environment in a body. One of the key life-limiting factors of the current component materials is the generation of numerous wear particles from the most widely used combination of artificial joint components, CoCr alloy on ultra-high-molecular-weight polyethylene (UHMWPE). The primary problems are wear and toxicity of wear debris which can lead to osteolysis and subsequent loosening of the implant. In particular, polyethylene particles activate macrophages which stimulate osteclasts, leading to bone resorption and loosening. Therefore, it is important to establish test methods for evaluating wear of materials under pseudo-physiological conditions and for evaluating toxicity of wear debris in-vitro by using cultured cells.

This project is a joint activity with TWA 1, Wear Test Methods. TWA 1 will conduct wear tests using CoCr alloy plates and UHMWPE pins. Following completion of the wear tests, TWA 7 will conduct cytotoxicity tests using the UHMWPE wear debris generated from the wear tests using a novel inverted cell culture method. This study will identify if a particular population of size, shape, or morphology is more bioactive than others. Interlaboratory comparisons of this technique will pave the way for future standardization.

Preliminary studies have been carried out on five different samples of polyethylene particles of varied size distributions from (a) pin on plate wear tests, (b) in-vivo samples, (c) a knee and hip wear simulator, and (d) commercially available polyethylene beads. Initial results based on SEM photographs and subsequent image analysis revealed that in-vivo wear debris were more spherical in shape and smaller in size than that from (a) and (c). The particles from (d) had the largest mean diameter. The cytotoxicity index decreased in the order in-vivo wear debris<knee simulator wear debris<hip simulator<pin-on-plate wear debris<polyethylene beads. It was concluded that the smaller size particles were phagocytised more effectively than those of larger size. The results also validated the inverted cell culture method as an effective method to study the phagocytosis of UHMWPE wear debris.

*Technical Working Area 10*

## COMPUTERIZED MATERIALS DATA

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There are currently two projects underway, Project No. 1, STEP Terminology Review, and Project No. 2, Generic Data-Sharing Platform.

STEP is an unofficial acronym (**ST**andard for the **E**xchange of **P**roduct model data) for ISO 10303 for the computer-to-computer exchange of technical information on manufactured products throughout their entire life cycle. STEP is intended to facilitate enterprise integration by simplifying computer systems integration no matter what type of computers are used or what languages are spoken by the persons in the enterprise. This is done by means of a standard called an application protocol (AP) which is a set of explicit rules that stipulates that the information must be sequenced in a specified way and the concepts being exchanged must be clearly defined and unequivocal by all parties involved. The objective of the STEP Terminology Review is to ensure consistency in definitions of entities and attributes among various STEP application protocols and to minimize to the extent possible the use during the exchange of technical-field-specific terms for identical concepts. The first application protocol selected is AP223, Aluminum Foundry. ISO TC184/SC4 has formally approved a liaison between STEP and VAMAS.

Databases of materials data are widely distributed all over the world. However, a common procedure to retrieve and use the data from these distributed databases does not exist because each database has its own format and structure. Guidelines for individual databases exist in ISO and in ASTM E49 especially for materials data. Further development of a generic platform for data exchange, without regard to the structure of the original database, is needed.

The objective of Project 2 is to clarify the prerequisite for developing a generic platform for electronic data-sharing systems of materials data. The project has three stages. Stage I is a feasibility study on electronic data-sharing platform for distributed materials data. Stage II will test the prototype 'data type definition' (DTD) template for existing databases. State III is the creation of the common template and common data-sharing platform. Stage I efforts are underway. Using the materials database framework from ASTM E49, including fatigue, creep, sintered metals, heat resistant metals, amorphous materials, the first draft of a referential DTD has been completed. The results can be found at the TWA 10 web site (http://www.nrim.go.jp:8080/vamas_twa10/).

*Technical Working Area 13*

## LOW CYCLE FATIGUE

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At present there is one activity underway. This VAMAS/EC project, Quantifying Data Uncertainties and the Validation of a Code of Practice for the Measurement of Bending in Uniaxial Fatigue Test Pieces, has now been completed. The objectives were: (a) to develop a framework for quantifying measurement uncertainties in low cycle fatigue data, (b) to validate the new Code of Practice for the measurement of bending caused by load misalignment in uniaxial fatigue testing, and (c) to provide recommendations for a best practice guide for routine low cycle fatigue testing of metallic materials. The final report for this project has been produced. Dissemination of the outputs from the project has continued through publications, oral presentations, and contributions to national and international standards development organizations. Findings from this work are making significant impact on the development of at least six new standards and a best test practice.

It had previously been reported that a new project on evaluation of residual stress measurement techniques had been started with specific emphasis on the XRD and hole drilling methods. A number of organizations have expressed interest in this proposed project. Plans are underway to circulate information on the current work objectives and plans to formulate a complementary research program on effects of residual stress on fatigue behavior.

## METAL MATRIX COMPOSITES

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The focus of this TWA is to develop an understanding of mechanical property determination on tensile test and fatigue test of metal matrix composites having heterogeneous structures at room and elevated temperatures and establish a unified and reliable testing method through a series of round robins. There are three active projects, each looking at a different metal matrix composite.

In Project No.1, preliminary fatigue data has been obtained for SiC whisker-reinforced aluminum alloy (SiC/A2009) tested at room temperature. Analysis of these data revealed the importance of specimen preparation, especially the need to eliminate all sharp corners and to polish the surface to reduce fatigue crack initiation sites. Final specimen preparation procedures and test parameters (200 °C) were selected and test specimens distributed to the participants.

A silicon carbide fiber-reinforced titanium alloy composite (SiC/Ti-15-3) is the test material for Project No. 2. The preliminary fatigue test data revealed a different fracture mechanism for small width specimens (2.5 mm) than for large width specimens (10 mm). Further analysis concluded that the wide specimens should be used. Therefore the wide width specimens should be tested in order to obtain the preliminary data. Following final fatigue testing at 480 °C, tensile test specimens will be distributed.

For Project No. 3 using fiber-reinforced $Al_2O_3$/Al metal matrix composite, nondestructive tests (C scan) of specimen uniformity and quality have been completed. Preliminary fatigue and tensile testing is underway. Following data analysis, final tensile and fatigue testing is planned for room temperature and 200 °C.

A questionnaire was distributed to current participants and others interested in metal matrix composites seeking suggestions for future projects. Many suggestions were received and new projects will be proposed based on future discussions within TWA 15.

*Technical Working Area 16*

## SUPERCONDUCTING MATERIALS

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There are currently five projects underway. Two draft standards have been produced for IEC/TC 90, Superconductivity, and a third draft standard will soon move into the balloting phase.

In Project No. 1, Bending Strain Effects on Critical Current in Oxide Superconductors, the objective is to establish a standard measurement method for the critical current in Bi-2212/-2223 oxide superconductors at 77 K with no external field. Preliminary measurements revealed that the critical current strongly depends on how the specimen is bent or deformed. To minimize ambiguity in the bending procedure, specially designed curved holders will be used for bending. Three kinds of Bi-2223 silver and silver alloy sheathed multi-filamentary tapes have been prepared for round robin testing. After completion of the round robin, a draft standard will be proposed to the IEC.

The objective of Project No 2, Measurement Method for Critical temperature in Oxide Superconductors, is to establish a standard measurement method. Round robin comparison testing was carried out using resistive, dc, and ac magnetization methods. Following analysis of the results, it was concluded that the resistive method should be recommended as the standard method. A draft IEC standard was prepared and proposed to IEC/TC 90 in early 2000.

In project No. 3, Measurement Methods for Trapped Field and Levitation Force in Bulk Oxide Superconductors, the objectives are to develop standard measurement methods of trapped flux density and levitation force. Preliminary studies were made on trapped flux density and levitation force measurement methods. During trapped flux density measurements, a problem with specimen breakdown occurred during the magnetization process. The problem was solved by impregnating samples with a resin that prevented the sample from cracking. A draft standard for the measurement of trapped flux density has been proposed to IEC/TC 90.

In Project No. 4, Measurement Methods for the Surface Resistance in Thin Film Superconductors, the objective is to validate an IEC draft standard, IEC 61788-7 on the surface resistance in thin film superconductors. A round robin test program was initiated with the goal of reaching an accuracy of 10 percent. In the initial round robin the data scatter was more than 10 percent and the error was traced to inhomogeneity in apparatus materials. Using a modified apparatus, a second round robin test program is currently underway. The draft standard IEC 61788-7 is now at the CD stage and will move to the CDV stage after refinement of the draft based on the results from the second round robin.

The objective of Project No. 5, Measurement Method for the Mechanical Properties of Oxide Superconductors is to establish standard test methods for mechanical properties such as yield strength and Young's modulus at room temperature. In the first round robin, participants will carry out the measurements using their own techniques and procedures. Following analysis of the results, test procedures will be formalized and a second round robin will be conducted using the specified testing parameters and conditions.

A new project has been proposed to clarify the relation between V-I characteristics and flux pinning, to determine the relation among irreversibility (critical) fields obtained by different measurement methods, and to establish an adequate and reliable measurement method.

# CRYOGENIC STRUCTURAL MATERIALS

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A draft of a new work item, Metallic Materials – Tensile Testing in Liquid Helium, was submitted to ISO/TC 164/SC1 in response to a request from SC1. The proposed draft was distributed to the members of the SC1 for voting.

Currently, there are three projects underway. Project No. 1, Interlaminar Shear Test on Graphite Fiber Reinforced Plastic (GFRP), is trying to develop an understanding of mechanical property behavior during interlaminar shear tests of GFRP at liquid helium temperature and establish a unified and reliable testing method through the use of round robins. Testing procedures with the advanced specimen geometry and specimens for the third round robin were distributed to the participants. The results were presented at the ICMC'99 meeting and the report will be submitted to the IEC as a Technology Trends Assessment document.

The objective of Project No. 2, Mechanical Tests in a High Magnetic Field, is to establish reliable methods of evaluating Young's modulus, yield strength, tensile strength, elongation, and fracture toughness at 4 K in a high magnetic field. Preliminary tests were carried out and the results indicated that the magnetic field had little effect on the load cell and/or extensometer. Titanium alloy specimens have been distributed to the participants.

Project No. 3, Advanced Fracture Toughness Test, is trying to develop an understanding and a J-integral testing method at liquid helium temperatures for a small-size round bar with a circumferential notch. Testing procedures and specimens for a round robin test program were distributed to participants. Tentative results were presented at the ICMC'99 conference.

## STATISTICAL TECHNIQUES FOR INTERLABORATORY STUDIES

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There are no current projects within TWA 18. The TWA had been inactive for the past several years and the new chairman assumed the position in 1999. The current task is the reorganization of the TWA as a body for statistical consulting and advice within VAMAS. The role of TWA 18 is to support the work of the other TWAs by offering advice and statistical consulting. The primary responsibility for the statistical planning and analysis should in most case reside with the statisticians from the organization organizing or coordinating a project. This is necessary to secure the necessary close contact to the scientists responsible in the field of application. Members of the TWA or statisticians from the coordinating organization should be involved in new projects from the initiation of the project.

The TWA will serve as a body of consultancy for specific questions and problems.

## MEASUREMENT OF RESIDUAL STRESS

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Currently TWA 20 is concerned with the measurement of residual stress by neutron diffraction. Four round robin samples are being examined. These include a shrink-fit aluminum alloy ring and plug assembly, a ceramic matrix composite, a shot-peened nickel alloy plate, and a ferritic steel weldment. These samples were chosen to represent a range of materials and practical applications of industrial importance. Measurements are almost complete. Analysis of the results on the ring and plug assembly is complete and the findings have been issued as a VAMAS report.

Interpretation of the measurements on the other samples is well in hand and should be presented at the next progress meeting. It is anticipated that all measurements will have been completed at that time. Considerable progress has been made in assembling the necessary information for preparing a draft standard for making measurements of residual stress by neutron diffraction. A protocol has been agreed to for obtaining reliable results. An outline document has been produced and a revised version will be prepared for discussion. When approved, it is intended that it will be proposed as an ISO Technology Trends Assessment document. Discussions have been held with ASTM and CEN concering the preparation of a standard. Further information can be found at the TWA web site: http:/www.risoe.dk/vamas-twa-20/

*Technical Working Area 21*

## MECHANICAL MEASUREMENTS FOR HARDMETALS

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The completed project on bend testing resulted in modifications to a documentary testing standard, ISO 3327, Bend Strength Tests for Hardmetals. The revised standard is now out for voting as a DIS with ISO member states, having been reviewed and approved at an ISO Committee meeting in 1999.

A new project has been formulated on toughness testing. The objective is to evaluate different toughness tests, including the Palmqvist indentation test. Following wide dissemination of the proposed project, a core group including at least 10 industrial laboratories from 8 countries has agreed to participate in the project.

The interlaboratory exercise will generate underpinning technical information on well characterised materials that will allow good practice for toughness tests for hardmetals to be specified. It will provide a wider understanding of the benefits of short bar/rod chevron notched beam (CNB) tests vis a vis single edged precracked beams (SEPB) and provide guidance on the allowable range for useful Palmqvist tests. It is anticipated that an ISO Technology Trends Assessment document will be proposed as a first step in recommending appropriate suitable test methods that have the wide acceptance of industry.

Participating organisations will be sent two sets of samples for Palmqvist tests, one already indented and one with as-ground surfaces. Participants will polish, indent and measure toughness by their own procedures on this second set as well as measuring the first set. In-house tests will be performed by appropriate organisations on three sets of material:

- Their own grades
- Two additional grades tested by everyone; to be selected
- One further grade, randomly selected.

A future project on the Comparison and Evaluation of Vickers and Rockwell Hardness Tests has been suggested as a result of confusion amongst hardmetal customers in Europe and North America where specifications may require different methods for hardness measurements.

# MECHANICAL PROPERTY MEASUREMENTS OF THIN FILMS AND COATINGS

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There are currently two active projects. Project No. 1, Measurement of Hardness and Young's Modulus of Thin Coatings Using Depth Sensing Indentation Instruments, has approximately 35 active participants. The objective is the development of methodologies for the determination of the mechanical properties of a coating in situ, and validation of the method by carrying out an international round-robin.

Progress in the analysis of the indentation loading-unloading curves has been made using the NPL spline fitting methodology. This provides a consistent method to determine the unloading slope required for calculation of sample stiffness (Young's modulus). The method has also been successfully applied to the analysis of data produced by an AFM for the determination of the indenter area function. Analysis of data provided by the participants using this methodology is now in progress. Although not all participants provided sufficient calibration data, it has been possible to agree on an approach for the data analysis. A final report is expected to be completed later this year. Information obtained in the project has already been incorporated into standards for depth sensing indentation being written by two ISO committees and two CEN committees.

The objective of Project No. 2, Adhesion of thin coatings, is to evaluate and compare test methods for adhesion of thin coatings (<10 μm), including bend testing and indentation, with the goal of developing a simple, quantitative engineering test. The work plan called for producing two TiN coating types that were well bonded and less well bonded to a stainless steel substrate. A mini-round robin was carried out using four-point bending to verify that the proposed testing method was applicable to the coated samples. The test relied on the generation of cracks whose spacing reaches a minimum when spallation occurs. The crack spacing can be related to the interfacial shear strength. As a result of the relatively strong adhesions of all of the TiN coatings, including those with 'poor' adhesion, the project is being redesigned.

A survey of possible new projects will be carried out to explore interest in residual stress measurement and other non-destructive test methods for thin film mechanical properties that might be initiated following the completion of Project No. 1.

# THERMAL PROPERTIES OF THIN CERAMIC FILMS AND COATINGS

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A multi-laboratory round robin to measure the thermal conductivity of thin ceramic films was carried out based on recommendations from the Workshop of Thin Film Thermal Conductivity held at the 13$^{th}$ Symposium on Thermophysical Properties in 1997. Thin film thermal conductivity is one of the parameters needed to predict the performance of electronic devices because of the use of insulating layers that can impede the flow of heat generated in active circuit elements to heat sinks. Round robin tests were used to evaluate measurement methods for determining thermal conductivity of thin silicon dioxide films on silicon wafer substrates. Silicon dioxide films were selected because silica is an important insulation material in devices.

Films of silicon dioxide on silicon were prepared with nominal film thicknesses of 50 nm, 100 nm, 200 nm, and 500 nm. Each participating laboratory chose its own measurement method. The methods included the three omega (3-$\omega$) method, thermal reflectance, photo-thermal beam deflection, photoacoustic method, thermal comparator, and the three-bridge method. Results from eleven laboratories were received. The analysis of the results is complete and a final report prepared.

Although eight different measurement methods were used, only the three omega method was used by multiple laboratories and thus a complete interlaboratory comparison was carried out only for the 3-$\omega$ method. Assuming that the thermal conductivity and interface thermal resistance are specimen independent and the interface thermal resistance is finite, the thermal conductivity for these silicon dioxide films was found to be $1.38 \pm 0.05$ W$\cdot$m$^{-1}$K$^{-1}$ at 20 $^{\circ}$C, very close to the handbook value of 1.37 for a interface thermal resistance of $(23 \pm 9) \times 10^{-9}$m$^2$K$\cdot$W$^{-1}$. Based on the 3-$\omega$ results it was concluded that: (a) interlaboratory reproducibility is good; (b) uncertainty increases with decreasing film thickness; (c) the interface thermal resistance cannot be ignored for thin films; (d) the special techniques required for specimen preparation make the procedure difficult to implement as a standard laboratory procedure; and (e) the conclusions should be validated for other specimen combinations.

No new projects are planned. A possible project to measure the thermal conductivity of thermal barrier coatings was suggested but there appears to be insufficient industrial interest at this time to develop the project further.

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