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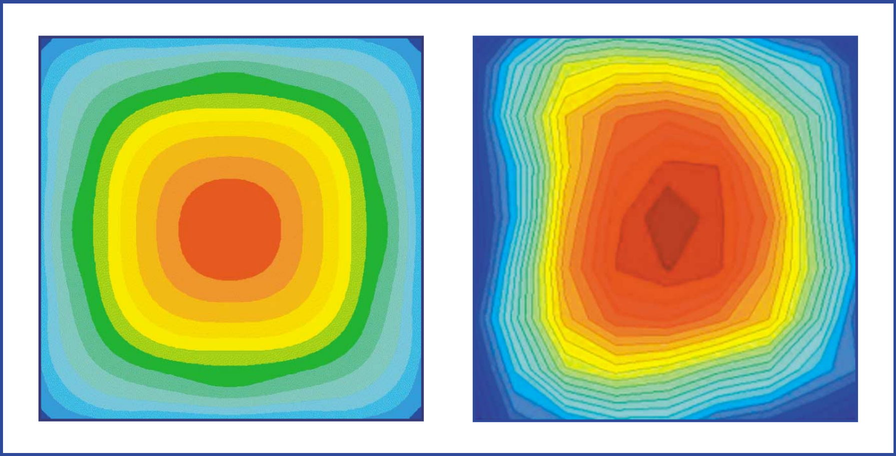

# BULLETIN NO. 25
## November 2002

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:*** Axial Stress Distribution in the Central Plane of a Water-Quenched 2024 Aluminum Bar
(610 mm x 75 mm x 75 mm)

***Left Image*** Calculated distribution using finite-element-methods
*(M. A. Newborn et al, ALCOA)*

***Right Image*** Measured distribution by neutron diffraction at NIST in conformance with guidelines developed under VAMAS Technical Working Area (TWA) 20
*The stress range is approximately 220 MPa tensile [red] to 220 MPa compressive [blue]*

***Photographs courtesy of:***

*Henry Prask, NIST Center for Neutron Research, Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Technology Administration, U.S. Department of Commerce.*

VAMAS

# • Table of Contents •

# • Chairman's Message •

I am both pleased and privileged to be able to report to you as the new Chairman of the VAMAS Steering Committee, having taken over from Steve Freiman, NIST. On behalf of the VAMAS community of scientists around the world, I would like to thank Steve and his colleague Jim Early, the immediate-past secretary of VAMAS, for their sterling work of leadership and organisation.

A major driver for science and research funding in this decade is, and will continue to be, the opportunities arising, coupled with a public fascination of, nanotechnology. Last year VAMAS collaborated with CENSTAR (the European standardisation and research steering body), to organise a two-day international workshop at the National Physical Laboratory, UK, on measurement needs for nano-scale materials and devices. The objective of the workshop was to identify measurement needs for themes in the area of nanotechnology in which the development of measurements and standards is critical to the commercialisation of new devices and components. The three themes selected and addressed were imaging at the nano-scale, nano-mechanical properties, and friction at the nano-scale. A full report on the workshop is presented later in this Bulletin. The outputs of the workshop have fed into the development of the new VAMAS Technical Working Area (TWA) on nanomaterials.

For those wishing to find out more about the latest VAMAS activities I encourage them to visit the VAMAS web-site (**www.vamas.org**) where details of the TWAs, and of new and current projects, plus VAMAS reports and the latest Bulletin can be found.

Finally, I would like to thank all the worldwide participants in VAMAS; the members of the Steering Committee, the chairmen of the TWAs and the scientists and technicians involved in the many and varied research activities, for their contributions to the undoubted success of VAMAS in achieving its eminently worthy objectives. I look forward to working with you over the coming years.

Colin Lea
Chairman

# • Feature Article •

## MEASUREMENT NEEDS FOR NANO-SCALE MATERIALS AND DEVICES
## Report of VAMAS/CENSTAR Workshop, 5-6 June 2002

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With acknowledgement to:
Dr S Freiman, NIST, Dr M Gee, NPL, and Dr T LeBrun, NIST
for their significant contributions.

### SUMMARY

As devices and device features shrink in size and grow in complexity, the measurement of their dimensions, features, geometry and properties presents major technical challenges. An inability to perform reliably these measurements presents a serious threat to the commercialisation of such devices. A successful, high-level international workshop, attended by leading experts in the field, was recently organised by the National Physical Laboratory (NPL), UK, and the National Institute of Standards and Technology (NIST), USA, on behalf of VAMAS1 and CENSTAR2. The objective of the workshop was to identify measurement needs for a range of themes in the area of nanotechnology in which the development of measurements and standards is critical to the commercialisation of new devices and components. The themes addressed were:

- Imaging at the nano-scale
- Nano-mechanical properties
- Friction at the nano-scale

A number of key issues within each of these themes were identified. For imaging the nano-scale, the trend for real-time, in-vivo measurements with analytical capability was highlighted. Furthermore, the importance of scanning probe tip characterisation was

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[1] **VAMAS:** The Versailles Project on Advanced Materials and Standards supports international trade through pre-normative research projects aimed at providing the technical basis for drafting codes of practice and standards for measurements on advanced materials. Its signatories are the G7 countries and the European Union.

[2] **CEN-STAR:** Standards prevent barriers to trade, provide technical support for legislation and promote competitiveness. CEN is the standardization body within the European Union. CEN-STAR is the CEN Standardization and Research Working Group tasked with establishing a more efficient link between European cooperative R&D and European standardization, improving the speed, quality and completeness of the standardization process.

viewed as critical to provide reliable and meaningful measurements. In the nano-mechanical properties theme it was highlighted that there is an overarching need for methods, standards, reference materials and guidelines in mechanical property determinations for the characterisation of nano-scale materials and devices. The most immediate high priority needs were for tests for a range of properties, including adhesion, deformation, stiffness and fracture. The overall requirements in nanotribology were considered to be the management of friction contact forces and the avoidance of wear. In nanoelectromechanical systems (NEMS) and micorelectromechanical systems (MEMS), the lack of control of friction is a "log jam" to many exciting new developments.

In summary, the workshop concluded that the top priority areas for attention were:

- Scanning Probe Microscopy (SPM) tip characterisation
- Development of best practice for SPM
- Modelling the imaging process / interpretation of the image
- Development of methods and standards for quantitative analytical characterization
- Development of nano-mechanical properties tests
- Development of scaling laws (to predict properties at the nano-scale based on data obtained at larger scales)
- Development of constitutive relationships for modelling friction at the nano-scale

(NB: The topics in this list were not ranked in order of priority by the workshop)

Pivotal to the success of such work is that it should be adequately funded and involve interdisciplinary team based projects where theory, fundamental experiments and nano-scale applications are coupled.

The outputs of the workshop, summarized above, have been provided to the EC as expert advice for consideration in their formulation of the Framework VI programme, and will also be used in the development of a new VAMAS Technical Working Area (TWA)[3] on nanomaterials to be led jointly by NPL and NIST.

## INTRODUCTION

Arising from a meeting between representatives of VAMAS and CENSTAR, a workshop to identify needs for measurement and STandardisation for nano-scale materials and devices was held at the National Physical Laboratory (NPL), UK on 5-6 June 2002. Hosted by NPL on behalf of VAMAS and CENSTAR the workshop was attended by representatives of National Measurement Institutes (NMIs), Universities and industry from Europe and the USA.

[3] VAMAS Technical Working Areas are technical committees that are multi-national in membership and are responsible for instigating and carrying out the research.

To set the scene for discussion the workshop commenced with invited presentations by leading international figures from NMIs, the EC and industry, and by leading international scientific experts on three specific topics, namely nanoscale imaging, nano-mechanical properties, and friction at the nanoscale (nanotribology). Dr Herrmann presented an overview of research into the nanoscale at Physikalisch- Technischen Bundesanstalt (PTB). Dr Saraiva Martins described forthcoming opportunities for funding of R&D through the Framework VI programme. Dr Hatto spoke on the need for a new technical committee (TC) in CEN on measurements at the nanoscale. He suggested that the TC could have four working groups covering physical properties, structural characterization, chemical composition and biological evaluation at the nanoscale. He commented that it may be possible to establish the TC within 1 – 2 years and have a work programme underway within 2 – 3 years.

### *Invited presentations at the workshop*

*Introduction to Measurement Needs for Nano-scale Materials & Devices*
*Dr Kamal Hossain, Director of Science & Technology, NPL, UK*

*Nanoscience and Nanotechnology in the EC Programmes*
*Dr Carlos Saraiva Martins, European Commission - Research DG*

*Measurement needs for nano-scale materials and devices*
*Dr Konrad Herrmann, PTB, Germany*

*The need for a new Technical Committee for nanoscale measurements*
*Dr Peter Hatto, IonBond Ltd*

#### *Imaging at the nano-scale*

*Scanning Tunnelling Microscopy: towards fast imaging in realistic environments*
*Prof. Joost Frenken, Leiden University, The Netherlands*

*Quantifying atomic scale composition: the 3-D atom probe and related techniques*
*Prof. George Smith, University of Oxford, UK,*
*Chairman of UK Institute of Materials Nanotechnology Panel*

*Measurements and Standards for Nanotechnology: Imaging and Length Metrology at the Nanometer Scale*
*Dr. Tom LeBrun, National Institute of Standards and Technology, USA*

#### *Nano-mechanical properties*

*Mechanical properties of materials at the nanoscale - an introduction*
*Dr David Mendels, NPL, UK*

*Friction at the nano-scale*

> *The scaling issue in friction measurement*
> *Dr Stephen Hsu, National Institute of Standards and Technology, USA*
>
> *Quantitative nanotribology with scanning probe techniques*
> *Prof. Robert Carpick, University of Wisconsin-Madison, USA*

Interspersed with the presentations were sessions in which the workshop participants broke-out into three focused sub-groups to formulate requirements for standards activities and pre-normative research in each of the three areas. Specifically, each group was asked to identify technology trends, barriers to progress, current measurements and standards activities, deficiencies in current measurements and modelling (particularly with an eye to future needs), and requirements for future standards, reference materials, and guidelines. Summaries of the findings of each of the sub-groups are presented later.

To conclude the meeting, a consensus view was reached on the top priority areas for R&D for the three topic areas. They were:

- Scanning Probe Microscopy (SPM) tip characterization
- Development of best practice for SPM
- Modelling the imaging process / interpretation of the image
- Development of methods and standards for quantitative analytical characterization
- Development of nano-mechanical properties tests
- Development of scaling laws (to predict properties at the nano-scale based on data obtained at larger scales).
- Development of constitutive relationships for modeling friction at the nano-scale

These areas were, however, not ranked in order of priority as such a ranking was considered inappropriate given the diverse range of interests of the participants.

## REQUIREMENTS FOR NANO-SCALE IMAGING (Dr T. LeBrun, NIST)

To identify near-term standards requirements for nanoscale imaging, the imaging subgroup of the VAMAS-CENSTAR workshop concentrated on widely used nanoscale imaging techniques such as scanning probe microscopy (AFM and STM), and electron microscopy (SEM and TEM). Other important techniques were also discussed including optical microscopy (near field and non-linear), ion-based microscopy (SIMS, field ion, atom probe), and x-ray microscopy (XPS, tomography), but the desire to identify important near-term needs and the expertise of the group focused the discussions on scanning probe microscopy (SPM) and electron microscopy.

## Technology Trends

The imaging subgroup identified a number of trends that can be classed into three groups:

- Measurements using real-world samples and environments
- Development of instrumentation to map multiple properties
- Customization of tools for specific experiments

*Measurements using real-world samples and environments*

The development of measurements using real-world samples and environments (c.f. UHV) is a particularly significant trend because it brings tools for understanding processes at the atomic and molecular level to practical problems. A striking example of this was given that showed atomic-resolution images of a platinum catalyst while it catalysed a burning reaction in a flow of reactant gases – a crucial window into a process (catalysis) that is still incompletely understood despite very extensive study and tremendous industrial importance.

But the importance of performing measurements using real-world samples and environments is much broader. Traditional laboratory experiments have focused on tractable systems such as perfect crystals in ultrahigh vacuum environments, and while these types of experiments have brought crucial understanding of fundamental principles, the application of these tools to real-world samples and environments brings us a great step closer to applying nanotechnology to meet important societal needs.

One extremely important example is biology and medicine. The ability to study the processes of individual cells at the molecular level (rather that the average behaviour of aggregates of cells) could profoundly change our understanding of cellular biology and hence our ability to cure disease. And this is exactly what new nanoscale imaging tools that operate in vitro or in vivo promise to provide.

In the industrial arena the ability to image samples over a range of pressures, temperatures, and chemical environments has opened the door to studying catalysis, fatigue, corrosion, and friction, to cite only a few important examples.

The specific trends toward real-world applications noted in discussion included measurements in-vivo and in-vitro, in reactive flows, and in high temperature/pressure environments. The trends in studying a wider range of samples also included samples with a range of heights rather than nearly flat surfaces, and complex materials such as biomaterials with complex interleaved structures or samples with ranges of hardness, conductivity and other properties.

*Mapping multiple properties*

A trend for multi-property mapping was identified by the sub-group:

Chemical characteristics: Concentration, chemical elements
Biological characteristics: Permeability, hydrophobicity/philicity
Electrical characteristics: Conductivity, capacitance
Materials properties: Hardness, friction, etc

*Customizing instruments for specific experiments*

- Combined instrumentation
  SEM + SPM to check tip quality, shape, degradation, control tip location
  NSOM + AFM for force and spectral information

- Designer tips and instruments
  Optimise tip geometry or functionalization to measure specific properties

- Diversity of applications and specialization
  Range of applications increasing rapidly, will drive need for customization
  Instruments will become more specialized

**Barriers**

The barriers to wider adoption of scanning probe microscopy principally concern the transition from research tools to production tools: making them easier to use, enhancing the performance, and getting the metrology right.

*Ease of Use*

Scanning probe microscopy is still a labour intensive activity; instruments require expert operators and results are frequently not reproducible. More automation is required to make scanning probe microscopy a productive and robust tool for industrial application. This includes automating set-up, operation, sample preparation and introduction, and calibration. Instruments also need to accept a wider range of samples, including that of sample size, sample roughness, and sample environment. As noted elsewhere in this report, the trend toward measurement in real environments is seen as one of the most important emerging capabilities of SPMs.

*Improved Performance*

Improving performance goes hand-in-hand with increasing ease of use. Drift is a key problem in maintaining reproducibility of measurements. The area of a sample imaged by an SPM typically drifts over time due to temperature variations, piezo hysteresis, and sources of long term drift. This limits the very important ability to track changes in a sample, and is a key contribution to measurement irreproducibility. While instruments with little drift (even when ramping temperature over a wide range) have been demonstrated in research laboratories, these capabilities must be extended to the wider community using commercial instrumentation.

One of the key barriers to wider application of SPM is that the technique has been largely qualitative in the past. To remove this obstacle calibration tools must be built into instruments. This includes calibration of scan range, linearity, and orthogonality of motion for all three axes, as well as calibration of electronic or spectroscopic measurements that may be used. Even more importantly the probe-sample interaction must be well understood.

Modelling the interaction of the probe and the sample is a key challenge that many nano-scale imaging tools share. In an SPM the shape of the tip determines the interaction with the surface, and therefore the image resolution and image aberrations — but tip shape is frequently not known. To make matters worse, the shape and chemistry of the tip can easily change during operation, leading to unpredictable changes in instrument performance. While algorithms and tools for in-situ tip characterization have been developed, they need to be more widely available in a standardized form.

Modelling is also centrally important for SEM imaging at the nanoscale, because the interaction of the e-beam with very small samples is complex and not completely understood. As features become smaller, adjacent areas of a feature can change the image of the part under study leading to a complex imaging process that can be simulated, but not simply inverted to allow correction of the image.

Another problem occurs because the electron beam in an SEM generally penetrates a sample more deeply than a few nm (penetration depths into silicon can easily exceed one micrometer under typical conditions). To restrict the sensitivity of the measurements to a smaller volume of the sample we must reduce the beam energy, but this changes the interaction with the sample — even up to the point of reversing the contrast in the image.

These interactions must be modelled and understood to allow SEMs to provide reliable imaging at the nanoscale.

### *Quantitative Mapping*

Finally, let us note in passing that while we have concentrated on the most frequently used imaging tools, other areas are rapidly developing such as spectroscopic imaging (both near-field and non-linear in the far field), and these will also have standards requirements to ensure reliable measurements.

## Current Measurements and Standards

Because nanotechnology science and industry is in it's infancy, standards needs are not yet well-defined but some related standards activities are already in progress. In dimensional metrology, instruments to measure step heights with atomic resolution have been developed, and steps toward using the crystal lattice as a natural length standard are underway. Note that this is not trivial because the best measurements on the silicon lattice parameter (the prime candidate for a lattice spacing artifact) are taken on some of the most highly perfect crystals ever fabricated, that have also been carefully characterized for isotopic concentration and for residual chemical constituents. The lattice parameter measurements are taken for the bulk, and corrected for these imperfections. While the correspondence between these measurements and the lattice parameter at the surface of an industrial grade silicon wafer may be close enough for many applications, the precise relation remains to be worked out.

To characterize tips algorithms such as the Blind Tip Reconstruction algorithm are available, and have been incorporated into at least one commercial instrument. Tip characterization artifacts are also available from commercial suppliers, but more standardization in this area would be helpful. Work is also underway to standardize performance metrics for positioning and motion control in SPMs. But this work is at an early stage. Finally, several groups have developed SPMs with interferometry to measure the motion of the probe or stage with high accuracy, but this work is largely restricted to research laboratories.

To model the SEM imaging process, Monte Carlo simulation packages are available, but this does not permit direct image analysis. Instead an array of reference structures must be simulated to produce a reference library that is compared to images. This work is underway and has been used by SEM customers with stringent requirements, such as the semiconductor industry.

**Measurement and Modelling Deficiencies and Requirements**

Identified issues were scored on the basis of perceived priority and the timescale on which that issue needed to be addressed, as follows:

| Priority | Timescale | |
|---|---|---|
| M | M | Guidelines for sampling |
| H | S | SPM tip characterization |
| H | S | Best practice for SPM<br>(use, calibration, estimation of uncertainty) |
| H | S | Modelling of imaging process (SEM, SPM, optimization) |
| M | M | Mechanical performance of SPM (inc. calibration artifacts) |
| M | M | Standards for instrument performance |
| H | S | Standards for quantitative analysis |
| H | M | Ease of use and interoperability |
| H | L | Designer tips to optimize instruments for experiments |

Note: Priority: H - high, M – medium and L – low
Timescale: S - short, M – medium and L - long

### Requirements for Future Standards, Reference Materials, and Guidelines

Many of the requirements for standards, reference materials and guidelines had been presented in earlier discussion (detailed above) and are therefore not repeated here.

## REQUIREMENTS FOR NANO-MECHANICAL PROPERTIES (Dr S Freiman, NIST)

Two primary technologies: (1) micro/nano electromechanical systems (MEMS/NEMS), and (2) high strength, nanotube composites were the focus of discussions held by this breakout group. However, the group felt that the measurement and standards needs were quite different for these two broad groups of applications. Furthermore, it was agreed that while films, having many important applications, were nanometre in extent on one scale, their primary use would be for other than mechanical properties and therefore the need for mechanical property data would take a lower priority.

### Micro/Nano ElectroMechanical Systems (MEMS/NEMS)

It was agreed that the particular applications of MEMS/NEMS that would be most in need of knowledge of mechanical properties were those in which moving parts were involved, i.e., switches and motors. The durability/reliability of such devices is currently a limitation to their widespread development. (Since nano-tribological issues are discussed separately, they will not be covered here.) There are currently no acceptable test methods to measure durability, and in fact, there are very few methods available to determine mechanical properties of any kind in this size range. While current kinds of tests, e.g. dogbone tensile tests, can be conducted, sample design and preparation is difficult, and obviously quite expensive. There are currently no models that would allow the reliable extrapolation of data from large-scale tests to predict the behaviour of material in this size range.

### Current Tests

Today, the most widely used direct tests for mechanical properties of small-scale devices involve nano-indentation (or nano-scratch) procedures. However, there are no widely accepted guidelines for the use of such procedures. Standards for nano-indentation measurements of hardness and elastic modulus are currently under development in VAMAS Technical Working Area 22. There is also ongoing work at the NIST Boulder Laboratories toward the development of very small-scale tensile tests for films, etc.

It was pointed out that, although they do not represent actual mechanical property measurements, there are both x-ray and laser acoustic methods for the determination of residual stresses. Residual stresses are expected to play a significant role in the reliability of nano-scale components.

### Needs/Priorities

Much of the discussion centred on the immediate and future needs for measurements and modelling in the nano-scale regime. The most immediate, high priority needs were for tests for a number of different properties. These properties include adhesion, deformation, stiffness, and fracture (not in any priority order). It was noted that a significant part of the problem in creating such tests are issues of locating, handling, and gripping specimens.

Also noted as a high priority need was the development of scaling law models that would permit the extrapolation of properties down to the nano-scale, taking into account the size and shape of the specimen. The group suggested that the creation of such models would be a longer-term goal.

An interest in the development of dynamic tests, and improvement of nano-scratch testing to improve accuracy and depth of penetration was also expressed.

## Nanocomposites

Because of the perceived manufacturing cost, the development of nanocomposites, i.e. composites in which the reinforcements are carbon nanotubes, for other than niche applications was viewed as being a very long-term issue. In fact, an ability for mass production of such composites was seen as the primary barrier that must be overcome if they are to be of economic importance. Nevertheless, it was noted that better test methods for such composites would possibly speed their development.

### Needs/Priorities

It was noted that one of the primary needs is for test methods that can be used to measure the interfacial strength between the carbon nanotube and the interface. It is this interfacial strength that is the major factor in determining the failure mode of the composite. Because of the small sizes of the nanotubes themselves, such a test must involve very low loads. In addition to specific interfacial measurements, determination of force and torque in the small size range will be needed. All of the above mechanical test needs were viewed as being high priority, short term, goals.

### Needs for Standards and Guidelines

It was noted that there is an overarching need for standards, reference materials, and guidelines in mechanical property determinations for nanotechnology. The highest priority need was indicated for a guideline, which summarized definitions, classifications, and terminologies relative to nanotechnology. There was also a desire expressed for reference materials for use in the tests under development, with a priority for nano-indentation procedures. Finally, it was suggested that testing guidelines for nanocomposites would also be quite valuable.

## REQUIREMENTS FOR NANOTRIBOLOGY (Dr M Gee, NPL)

The overall requirements in nanotribology were considered to be the management of friction contact forces and the avoidance of wear. Another way of looking at this need is to be able to provide the required "designer" friction through the control of surface energy and chemistry.

In NEMS/MEMS, the lack of control of friction was considered a "log jam" to many exciting new developments. Thus without better control, commercial applications for NEMS/MEMS would be restricted to applications where there were no moving surfaces in contact due to reliability and durability issues, severely restricting commercial development. Nevertheless, areas where developments are likely on the medium term are in actuators, sensors, and process control. NEMS/MEMS was seen to be a pervasive technology with these types of devices becoming very common in all aspects of commerce, industry and society. One of the key drivers in the next few years was considered to be the development of technologies that allowed all of the different types of NEMS/MEMS technology together with computer elements to give complete integration of devices on a single chip.

Hard disk technology is a major current commercial application where there are continued nanotechnology developments where nanotribology is crucial. An interesting new application was the development, initially by the US military, of micro motors that were intended for use as replacements for batteries. These would be used to power the electronics of the "soldier of the future", addressing the reliability and supply issues of batteries.

Nanostructured high hardness, low friction coatings are already in commercial production, and it was expected that there would be a huge growth in this area in the next few years. Applications include coatings for tools, gears and engine parts. This was driven by the dramatic improvements in functional performance possible through technologies that gave nanostructure in the coating through, for example, MBE processing using functional atom clusters, or the use of multiple very thin layers of different chemical composition and structure. One possible breakthrough technology would be the development of coatings that allow dry cutting, that is, the elimination of cutting fluid for machining operations. These fluids are toxic and harmful to the environment. Eliminating the use of these fluids would have a large environmental, health and economic impact. This requires cutting tools that are sufficiently strong and tough to machine in dry conditions, necessitating an understanding of dry friction and wear.

In the biological application area there were two opposed areas where control of friction on a nanoscale was perceived to be important. These were the need for no friction and zero biological interaction for instruments such as catheters, and the need to promote adhesion and the interaction with biological material for many implant and tissue engineering applications.

A final area where nanotribology was seen to be important in the long term was the interaction of elements in micro/nano assembly devices.

## Barriers

The barriers to efficient developments in nanotribology were seen to be the

- Lack of metrological tools
- Lack of appropriate models for friction.
- Lack of analysis tools
- Lack of real time tools
- The lack of methods for the measurement of friction at nanocontacts under conditions realistic to final applications.
- The lack of high quality literature in the area combined with a morass of low quality literature.
- The lack of force and displacement sensors with a combination of high sensitivity, accuracy, and appropriate and controllable stiffness.

## Current Measurements

Currently, many measurements are carried out using AFM or other forms of SPM. This instrument gives nm resolution in all three axes and force resolution in the pN to nN range. Applied stresses range from MPa to GPa. Many measurements are also performed with surface force apparatus. This instrument gives very high resolution and sensitivity in force and displacement measurement in the direction normal to the contacting surfaces, but the dimension of the contacts in the x-y direction is much larger ($\mu$m). The force resolution is in the $\mu$N to mN range. Applied stresses range from kPa to 10's of MPa

Many laboratories have also developed their own custom devices for specific measurements. The study of the performance of nm scale thickness layers of lubricants has been revolutionized by devices based on interferometric measurement.

Nanoindenters are used in many studies of the mechanical response of materials to nanoscale contacts, and in many cases have been modified to enable nanoscratch tests to be performed. Specific test systems for nanoscratch testing are also available.

There are no specific standards for testing in nanotribology, but recognized calibration methods are emerging through the instrument manufacturer and user communities. It was noted that calibration practices amongst AFM researchers are not well-standardized nor uniformly practiced.

Another suggestion was the adoption by journals that publish nanotribology work of expected standards for calibration, since the methods are available but not widely or uniformly used.

## Deficiencies and Requirements

The issues identified were scored on the basis of priority and timescale on which that issue needed to be addressed. Please note that in the assessment of timescale, an S marking is given for a short-term need that nevertheless may only have a solution in the longer term. In some cases two sets of marks were given, e.g. the speed of performing experiments was seen to be of medium short-term importance, and high long-term performance.

| Priority | Timescale | Aspect |
|---|---|---|
| H | S | Lack of nm (traceable) calibration artifacts for: height, x & y displacement, force |
| H | S | Tip shape measurement and calibration |
| M | M | Tip chemistry: control, fabrication |
| M | S | Force and displacement measurement and control |
| H | S | Better force sensor design, including: servo, drift, stiffness, sensitivity, x,y,z decoupling, lateral force/normal force decoupling |
| M | S | Provision of variable temperature environment |
| H | S | Environment (gas species, pressure) |
| M<br>H | S<br>L | Speed of performing experiments |
| H | L | Molecular dynamic modelling: appropriate potential, extend time scale, extend simulation size (# of atoms) |
| H<br>L | S<br>L | Extend classical modelling of contacts |
| H | S | The need for a fundamental understanding of the science of nanotribology - identifying the individual components of friction (the constitutive laws) |
| H | S | Surface chemistry: understanding, dynamics |

Priority: H- high, M – medium and L – low
Timescale: S- short, M – medium and L - long

## Standards, Reference Materials, and Guidance

The most important STandardisation requirement was thought to be the supply of nm scale calibration artifacts. There was also a need for methods for the calibration of force and displacement, but it was considered that this was often best done through the calibration of the full force-displacement response of an instrument.

The measurement of the tip shape was considered an immediate need that had a major effect on all aspects of nanotribology.

The group thought that a simple way of driving measurement in nanotribology forward was through the provision of practical guidance for users. In time these documents might progress to formal standards.

## ACKNOWLEDGEMENT

The workshop organizers are pleased to acknowledge and are very grateful for the contributions of all the attendees of the workshop and in particular to Dr S Freiman, NIST, Dr M Gee, NPL, and Dr T LeBrun, NIST who were chairmen of the breakout group sessions and authored much of the information presented above.

# • Feature Article •

## STATUS OF VAMAS TECHNICAL PROJECTS

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As part of an expanded outreach effort by the VAMAS Secretariat, a number of new reports have been produced in the past few years to highlight the role and impact of VAMAS pre-standardization or pre-normative technical activities. Reflecting the cumulative work of the many VAMAS technical committees or Technical Working Areas (TWAs), these documents include: **VAMAS Publications – 1983 to the Present** (a bibliography of technical papers published in archival journals, conference/symposium proceedings and VAMAS technical reports, issued annually); **VAMAS Technical Working Area Annual Reports** (a summary of TWA technical activities, issued annually); **VAMAS Contributions to Standards Development** (a list of draft, revised, and/or new standards, standard reference materials and Technology Trends Assessment (TTA) reports based all or in-part on VAMAS technical contributions, issued annually); and **VAMAS Technical Projects** (a list of current and recently completed technical projects, issued annually). Most of these documents are available in hard copy format as well as being posted on the VAMAS website, (www.vamas.org).

The following Table is the list of current projects as of June 2002, projects completed or discontinued during 2001-2002, and projects completed during 2000-2001 and 1999-2000.

[For each TWA in the table, the first column identifies the Project Number and the second column identifies the Project Title.]

### CURRENT VAMAS PROJECTS [JUNE 2002]

| TWA 1 | **Wear Test Methods** |
|---|---|
| 01 | Compilation of wear test standards |
| 02 | Ball cratering wear testing |
| 03 | Wear debris characterization methods and representation {with TWA 7} [currently on hold] |

## TWA 2 Surface Chemical Analysis

| | |
|---|---|
| 13 | Tests of algorithms for data processing in AES - Factor analysis and intensity |
| 14b | Tests of algorithms for background subtraction in AES |
| 14d | Tests of algorithms for angle-resolved XPS |
| A3 | Interlaboratory study of static SIMS repeatability and reproducibility |
| A6 | Evaluation of uncertainties in XPS peak intensities associated with different techniques & procedures for background subtraction |
| A7 | Evaluation of electron beam damage of $SiO_2$/Si in Auger microprobe analysis **[new 2001-02]** |

## TWA 3 Ceramics for Structural Applications

| | |
|---|---|
| 14 | Determination of phase composition and percent crystallinity in hydroxyapatite |

## TWA 5 Polymer Composites

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|---|---|
| 01 | Assessment and recommendation to ISO on Mode II fracture test methods |
| 02 | Measurement of mechanical properties for the fibre-matrix interface |
| 03 | Measurement of through-thickness testing properties |

## TWA 7 Biomaterials

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|---|---|
| 01 | Evaluation of cytotoxicity of UHMWPE wear debris {with TWA 1} [currently on hold] |

## TWA 10 Computerized Materials Data

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|---|---|
| 01 | STEP terminology review |
| 02 | Generic data-sharing platform |

## TWA 15 Metal Matrix Composites

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|---|---|
| 01 | Develop reliable tensile test and fatigue test methods for MMCs at room & elevated temperatures - SiCw/A2009 MMC |
| 02 | Develop reliable tensile test and fatigue test methods for MMCs at room & elevated temperatures - SiC/Ti-15-3 MMC |
| 03 | Develop reliable tensile test and fatigue test methods for MMCs at room & elevated temperatures - $Al_2O_3$/Al MMC |

## TWA 16 Superconducting Materials

| | |
|---|---|
| 01(wg1-1) | Bending strain effects on critical current in oxide superconductors |
| 02(wg1-2) | Measurement method of critical temperature in oxide superconductors |
| 03(wg2-1) | Measurement methods for trapped field and levitation force in bulk oxide superconductors |
| 04(wg3-1) | Measurement methods for the surface resistance in thin film superconductors |
| 05(wg4-1) | Measurement method for the irreversibility field in oxide superconductors |
| 06(wg1-3) | Measurement method of irreversibility field in oxide superconductors **[new 2001-02]** |
| 07(wg1-4) | Coupling loss measurement in multifilamentary HTS superconductors **[new 2001-02]** |

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|---|---|---|
| **TWA 17** | **Cryogenic Structural Materials** |
| 05 | Interlaminar shear test on GFRP |
| 06 | Mechanical tests in high magnetic field |
| 07 | Advanced fracture toughness test |

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|---|---|
| **TWA 21** | **Mechanical Measurements for Hardmetals** |
| 02 | Toughness tests for hardmetals |

| | |
|---|---|
| **TWA 22** | **Mechanical Property Measurements of Thin Films and Coatings** |
| 01 | Measurement of hardness and Young's modulus of thin coatings using depth sensing indentation instruments |
| 02 | Adhesion of thin coatings |
| 03 | Elastic properties of thin films and coatings **[new 2001-02]** |

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|---|---|
| **TWA 24** | **Performance Related Properties for Electroceramics** |
| 01 | International intercomparison of direct piezoelectric coefficient using the Berlincourt method |
| 02 | Measuring $d_{33}$ from displacement of electric field strength **[new 2001-02]** |

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|---|---|
| **TWA 25** | **Creep/Fatigue Crack Growth in Components** |
| 01 | Creep/fatigue crack growth in components |

| | |
|---|---|
| **TWA 27** | **Characterization Methods for Ceramic Powders & Green Bodies** |
| **01** | Determination of coarse particle fraction in ceramic powders |

| | |
|---|---|
| **TWA 28** | **Quantitative Mass Spectroscopy of Synthetic Polymers** |
| **01** | Method development for MALDI TOF MS of low molecular mass polystyrene **[new 2001-02]** |

## VAMAS PROJECTS COMPLETED DURING 2001-2002

| | |
|---|---|
| **TWA 2** | **Surface Chemical Analysis** |
| 02 | Development of calibration data for the energy scales of Auger-electron spectrometers |
| 03 | Procedures for quantitative X-ray photoelectron spectroscopy |
| 05 | Development of reference materials prepared by ion implantation |
| 21 | Tests of algorithms for the analysis of multicomponent spectra in XPS |
| A2 | Evaluation of static charge stabilization and determination methods in XPS on non-conducting samples |
| A4 | Evaluation of multilayer reference coatings for quantitative GDOES depth profiling |
| A5 | Interlaboratory study of the degradation of organic materials in XPS analysis |

**TWA 3 Ceramics for Structural Applications**

13 High temperature flexural strength

**TWA 13 Low Cycle Fatigue Superconducting Materials**

01 Quantifying data uncertainties and validation of a code of practice for the measurement of bending in uniaxial fatigue test pieces

**TWA 20 Measurement of Residual Stress**

01 Measurement of residual stress by neutron diffraction

## VAMAS PROJECTS DISCONTINUED DURING 2001-2002

**TWA 2 Surface Chemical Analysis**

A1 Use of the infinite velocity method for SIMS quantification

## VAMAS PROJECTS COMPLETED DURING 2000-2001

**TWA 2 Surface Chemical Analysis**

30 Development of a common data processing system for AES and XPS

**TWA 3 Ceramics for Structural Applications**

12 Fracture toughness by the SEVNB method

**TWA 23 Thermal Properties of Thin Ceramic Films and Coatings**

01 Thermal conductivity of ceramic films

## VAMAS PROJECTS COMPLETED DURING 1999-2000

**TWA 2 Surface Chemical Analysis**

09 Intercomparison of Auger electron intensity measurements

14c Tests of algorithms for quantitative XPS by peak shape analysis

23 Absolute calibration of XPS instrument intensity scales

**TWA 21 Mechanical Measurements for Hardmetals**

01 Bend testing of hardmetals

## WEAR TEST METHODS

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Efforts to find a consortium of laboratories for the calibration of a force sensor in the nano-Newton range proved to be a difficult challenge. The users of Atomic Force Microscopes (AFM), who need spring constants for the AFM-cantilevers, use many different types of instruments that are not compatible with each other. At the present time, these differences in the types of instruments and how they are used have made it difficult to define a joint project.

**Status of current Projects**

At present there are three active projects.

The objective of Project No. 1, Compilation of Wear Test Standards, is to provide a validated source for standardized wear test methods that can be used by engineers and tribologists. An NPL report containing a compact disc with the database [NPL Report MATC(A)07, September 2001] is available. No further progress has been reported.

The goal of Project No. 2, Ball Cratering Wear Testing is to establish this method as a standard and to produce a recommended test procedure. The draft procedure has been written as a draft CEN and a draft ASTM Standard and will be presented to the relevant standard committees shortly. Funding has been gained from the EU (Project CRATER) to support the standardization of this test method. An interlaboratory exercise with 10 participating laboratories is currently in progress

Project No. 3, Wear Debris Characterization Methods and Representation, (in conjunction with TWA 7) remains on hold due to the temporary interruption of TWA 7.

## SURFACE CHEMICAL ANALYSIS

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Since its inception, TWA 2 has initiated 42 TWA projects. Seven projects were completed in the past year. Twenty-five projects had previously been completed and there are four inactive projects that could not be completed for lack of resources. The 6 current active TWA projects are listed below.

| No. | Project Title |
|---|---|
| 13. | Tests of algorithms for data processing in AES - Factor analysis and intensity |
| 14.* | (b) Tests of algorithms for background subtraction in AES |
| | (d) Tests of algorithms for angle-resolved XPS |
| A3. | Interlaboratory study of static SIMS repeatability and reproducibility |
| A6. | Evaluation of uncertainties in XPS peak intensities associated with different techniques and procedures for background subtraction |
| A7 | Evaluation of electron beam damage of $SiO_2$/Si in Auger microprobe analysis |

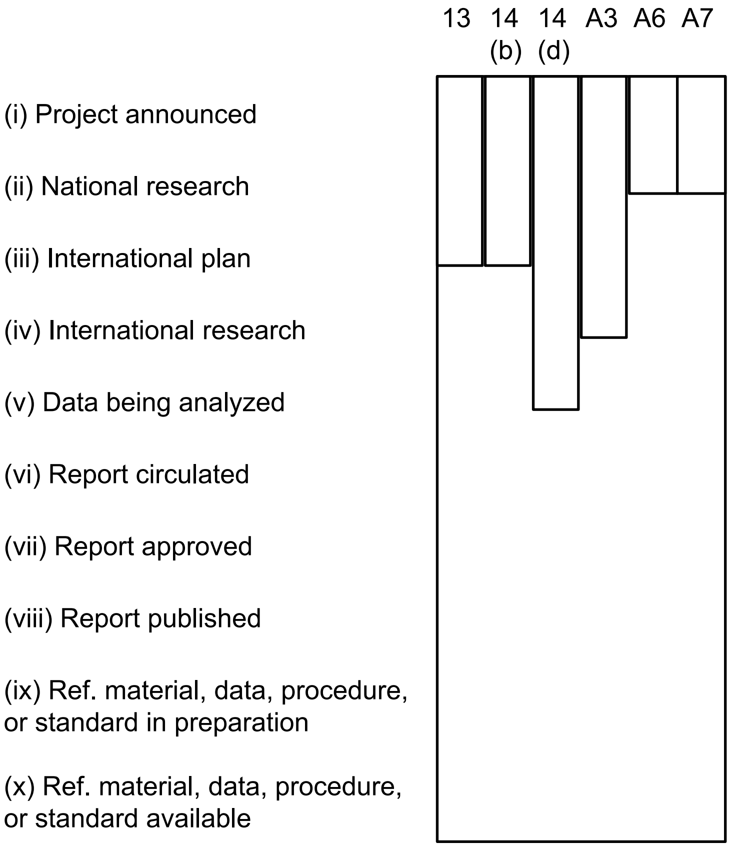
*This project received support from the Community Bureau of Reference, EC.

The states leading active TWA 2 projects are as follows:

| | | | |
|---|---|---|---|
| Germany | 13 | USA | A6 |
| Japan | A7 | EC | 14(b), 14(d) |
| UK | A3 | | |

Many of the outputs of TWA 2 projects have been incorporated or are in the process of being incorporated into ISO standards. Table 1 identifies the standards or draft standards of ISO Technical Committee 201, Surface Chemical Analysis, arising from or related to TWA 2 projects. ISO/TC 201 currently has eight subcommittees and seventeen working groups. The VAMAS TWA 2 is an official liaison body (category A) with ISO/TC 201 and seven of its subcommittees. Effective communication between ISO/TC 201 and VAMAS TWA 2 is enhanced by the fact that the chairman and vice-chairman of VAMAS TWA 2 and some of the VAMAS TWA 2 project leaders hold positions as chairmen of ISO/TC 201 subcommittees, conveners of ISO/TC 201 working groups, or as experts on ISO/TC 201 working groups.

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




During the past year, TWA 2 projects 2, 3, 5, 21, A2, A4, and A5 were successfully completed. The TWA 2 Chairman is extremely grateful for the efforts of the leaders of these projects and the project participants in bringing these projects to very satisfactory conclusions. In Project 2, needed data for the calibration of the energy scales of Auger electron spectrometers were acquired. This work and related work on the calibration of binding-energy scales of X-ray photoelectron spectrometers was performed predominantly at NPL and to a lesser extent at NIST. Detailed procedures were developed for these calibrations that have led to one published ISO standard, two ISO draft international standards that are expected to be published shortly as ISO standards, and one ASTM standard. Users establish control charts that show whether a particular instrument is "in calibration," that is, whether it is operating within the tolerance limits set by each analyst for the needs of the analytical work.

In project 3, extensive investigations have been performed to establish electron-transport models and to provide data for film-thickness measurements by Auger-electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS). This work has culminated in the development of an algorithm for the calculation of so-called practical electron effective attenuation lengths (EALs) and the release of a NIST database with which analysts can conveniently obtain practical EALs for materials and measurement conditions of interest. These practical EALs can then be used to obtain thicknesses of overlayer films from measured AES or XPS intensities.

In project 5, a detailed measurement procedure has been developed for calibrating the concentrations of dopants implanted into silicon for semiconductor-device fabrication. This work has led to a draft ISO Technical Report. The project leader, Dr. W. Gries (formerly at Deutsche Telekom and now retired) plans to publicize the results of the work in other ways.

In project 21, algorithms in three available software packages for the analysis of multi-component spectra in XPS were tested. This work showed the existence of unsuspected systematic errors in some algorithms. The test data used in this analysis are now available on the NPL web site.

In project A2, an interlaboratory comparison was conducted to evaluate the use of 15 nm gold particles for surface-charge stabilization procedure in XPS measurements on non-conducting specimens. The surface charge has to be stabilized in such measurements in order to identify chemical state reliably. Twenty-seven laboratories participated in this comparison that was led by the Bundesanstalt für Materialforschung und –pruefung (BAM) in Germany. This work and the importance of the problem have now led to the development of a draft ISO/TC 201 document, now at the Committee Draft stage, on the reporting of methods for charge control and charge correction in XPS.

In project A4, an interlaboratory comparison was conducted to evaluate multilayer reference coatings for quantitative glow-discharge optical-emission-spectroscopy (GDOES) depth profiling. GDOES measurements and other characterizations were performed on Ti/Al and $SiO_2/TiO_2$ test samples. Thirty-six laboratories participated in the GDOES intercomparison that was also led by BAM. A detailed report of the intercomparison has now been published.

In project A5, an evaluation was made of a procedure to determine the rate of degradation of three polymers due to X-ray irradiation during XPS analyses and to estimate the X-ray flux during an XPS analysis. Forty laboratories participated in an interlaboratory comparison led by the National Institute for Materials Science in Japan. Two polymers, poly(vinylchloride) and a blend of nitrocellulose and cellulose acetate, were found to be suitable materials for comparisons of X-ray fluxes in different XPS instruments. A report of this work has just been published.

Project A1 was discontinued during the past year due to a lack of sufficient resources for its continuation.

Satisfactory progress has been made in the remaining active TWA 2 projects during the past year. A new project, project A7, was initiated to determine the rate of electron-beam-induced damage to thin films of silicon dioxide on silicon during Auger microprobe analyses. Several interlaboratory comparisons are planned or are underway and, in other projects, improved scientific infrastructure and measurement capabilities are being developed.

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 | WD 16268: Ion-implanted surface-analytical reference materials |
| SC3: Data Management and Treatment | ISO 14976: Data transfer format<br>ISO 14975: Information formats |
| SC4: Depth Profiling | ISO 14606: Otimization of sputter depth profiling<br>TR 15969: Measurement of sputtered depth |
| SC5: AES | CD 18118: Guide to use of experimental relative sensitivity factors for the quantitative analysis of homogeneous materials (AES & XPS) |
| SC7: XPS | ISO 15472: Calibration of energy scales (XPS)<br>DIS 17973: Calibration of energy scales for elemental analysis (AES)<br>DIS 17974: Calibration of energy scales for elemental and chemical state analysis (AES)<br>CD 19318: Reporting of methods used for charge control and correction (XPS)<br>WD 18327: Unintended degradation by X-ray photoelectron spectroscopy |

| ASTM E42 Subcommittee | Title of standard or draft standard |
|---|---|
| .03: AES & XPS | E2108-00: Calibration of electron binding-energy scale of X-ray photoelectron spectrometer |
| .08: Ion Beam Sputtering | E1636-94: Describing sputter-depth profile interface data |

# CERAMICS FOR STRUCTURAL APPLICATIONS

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Currently there is one active project in the working area. This is a round robin on Determination of Phase Composition and Percent Crystallinity in Hydroxyapatite organized by Dr. Karin Hing, IRC in Biomedical Materials, Queen Mary and Westfield College, London, UK. Participating in the project are: Monash University, Australia, Eindhoven University of Technology, Nederland, Queen Mary and Westfield College, London, UK, National Institute of Standards and Technology, USA, University College London, UK, Federal Institute for Material Research and Testing, Germany, Kyocera Corporation, Japan, Japan Fine Ceramics Center, Japan, National Physical Laboratory, UK, Europese Unie – Petten, Nederland, ENEA - Divisione Nuovi Materiali, Italy, Queensland University of Technology, Australia, Cranfield University, UK, Kyoto University, Japan, Ceram Research, UK, National Institute of Advanced Science and Technology, Japan, and ENEA, New Materials Division, Italy.

A project on measuring the compressive strength of ceramic matrix composites has been proposed by Japan Fine Ceramics Center. However, there have been problems securing a broad international participation, and this project is not yet approved by the steering committee.

Coordination with ISO TC 206, Fine Ceramics is still good at the current B liaison level. The test method of measuring fracture toughness by the SEVNB method (project 12) has been proposed as a CEN method under TC 184. The draft goes out for balloting this year, and could be available as a standard in 2003. Coordination with ASTM C28 Ceramics is also very good.

## Status of Current Projects

### Project 14. Measurement of crystallinity and phase composition of hydroxyapatite by XRD

Hydroxyapatite (HA) is a ceramic material increasingly used as a biocompatible monolithic material or a coating encouraging adaptation of an implant into the human body, but the body's response depends critically on the phase composition of the material. In particular as small changes in Ca:P ratio (and hence phase composition) have been demonstrated to have a profound effect on the biological response they elicit in vivo, it is important that standard methods of analysis and reporting are employed to ensure that all data are comparable. The objective of this round robin is to assess the level of accuracy and repeatability achievable by a new method proposed as an ISO standard for the quantification of HA crystallinity and phase composition. This new method differs from the usually cited method and potentially removes uncertainties stemming from overlapping peaks.

The XRD pattern for HA has many peaks in the range from about 7° to about 60° 2θ CuKα. The most intense peaks are in the range 30° to 35°, which coincides with the peak of the amorphous band if amorphous calcium phosphates are also present. Impurity phases such as α- and β-tricalcium phosphate (TCP), even CaO, can also be present and must be controlled at low levels for appropriate bioactivity of the product. To measure these at low levels in HA means that the peaks must be identified, separated and background stripped. The minor impurity phase is very close to the main HA peaks, and is difficult to separate effectively. In addition, there can be an amorphous background from uncrystallized or poorly crystallized HA. The main peak is centered under the principal lines of HA and of the impurities. If these peaks are to be used for the estimation of phase content, this background has to be both estimated and removed, which is difficult to do in a reliable fashion.

The method being proposed to ISO, and being tested out in the round robin, is to use a different part of the XRD pattern to perform the calculations, specifically the 39°-52° 2θ portion. In this region, amorphous calcium phosphate displays a second band of amorphous scatter, but of much lower intensity. There are no significant impurity peaks present in this band. Given that impurities are typically present as less than 5% of the total crystalline phases present in the sample, choosing this region significantly reduces the potential for inaccuracies due to over lapping of peaks characteristic of different phases, and removes the necessity to deconvolute the pattern.

Participants in the round robin received quantities of pure HA, a calcium phosphate glass, α- and β-TCP and CaO as the reference powders for calibrations. A total of 10 combinations of these materials were mixed up and the X-ray patterns determined to provide the calibrations needed. Participants then prepared XRD traces of the sample powders in a consistent manner over the range of at least 20° to 55° 2θ in order to provide the zones for analysis. Analysis of the calibration samples provides the calibration parameters that are then used to estimate the phase proportions in the unknowns. Data are returned using a spreadsheet system, together with experimental details, particularly of the software systems employed, and any experimental difficulties encountered.

This round robin is not yet complete. However, so far, the reproducibility of the method appears good and the method appears able to reliably determine whether impurity levels are in excess of, or below 5%. However, in performing the data analysis it is clear that the method could be further improved (generally by simple changes to the calibration calculations), and to this effect, some of the participants have suggested a number of changes to the method to improve precision of the analysis. The organizer will assess these changes in turn and draft an improved methodology for the final report. The organizer will evaluate and compare the sets of results and identify the consistency of the results obtained in order to provide a critique of the method for the ISO Committee.

Two International Standards for hydroxyapatite ceramics and hydroxyapatite coatings are currently at the committee draft stage of ISO/TC150/SC1. The purpose of these two documents is to specify the key characteristics that define hydroxyapatite materials and to stipulate the requirements that shall be met to allow their use as materials for surgical implants.

At the meeting of ISO/TC150/SC1 in Singapore, December 1997, the working group on hydroxyapatite proposed that in order to avoid delay in publication of the specifications detailed in parts 1 and 2, the methods of test should be published as separate documents. This approach was deemed necessary because:

(a) There are many different methods currently employed to characterize these materials.
(b) The results of chemical characterization can be highly method dependent.
(c) Some methods require a high degree of operator skill and experience to achieve acceptable levels of accuracy and repeatability.

These issues are particularly sensitive as small changes in Ca:P ratio (and hence phase composition) have been demonstrated to have a profound effect on the biological response they elicit in vivo. Therefore it is important that standard methods of analysis and reporting are employed to ensure that all data are comparable.

At the meeting of ISO/TC150/SC1 in London, October 1998, the working group on hydroxyapatite agreed that selection of a suitable method of test (in terms of both ease of use and quality of results) for determination of the phase purity and crystallinity of hydroxyapatite coatings posed a considerable problem due to the variety (and sometimes complexity) of the methods reported in the literature. It was agreed that the most appropriate method (in terms of best accuracy for least complexity) should be selected and subjected to an international round robin to assess the suitability of the method to the application. In order not to slow progress of the publication of part 3 of ISO 13779-3 Implants for Surgery - Hydroxyapatite - Chemical Analysis and Characterization of Crystallinity and Phase Purity a method was drafted and sent to ISO in June where it was published as a committee draft. The X-ray method employed is new and potentially more reliable, and is designed to avoid some of the problems associated with some of the existing reported methods employed.

Relevant Standards

ISO/DIS 13779-1 Implants for Surgery – Hydroxyapatite – Part 1. Ceramic Hydroxyapatite.

ISO/DIS 13779-2 Implants for Surgery – Hydroxyapatite – Part 2. Coatings of Hydroxyapatite.

ISO/DIS 13779-3 Implants for Surgery – Hydroxyapatite – Part 3. Chemical Analysis and Characterization of Crystallinity and Phase Purity. ISO/TC150/ Committee draft.

## Proposed Future Projects

A Round Robin on Compression Tests For Ceramic Matrix Composites has been proposed by Dr. Mineo Mizuno, Japan Fine Ceramics Center, Nagoya, Japan.

### Background

Ceramic matrix composites (CMC) exhibit non-brittle behavior, and have been applied to structural components used in aerospace and other aggressive environments. The mechanical response, however, differs from typical ceramics and also from other ductile materials such as metals and polymers. Compression strength of CMCs is relatively important in designing CMC components because the compression strength is not as great as that of monolithic ceramics. The objective of this round robin is to assess the level of reproducibility and repeatability achievable by a method proposed for the determination of compression strength of CMCs. In particular, the round robin is planned to focus on the bending contribution during loading and on the friction due to vertical supports for test specimens, and aims at revealing key techniques in applying uniform compression stresses to avoid buckling in measuring compression strength. A continuous SiC fiber composite in a SiC matrix will be supplied to participants for measurements of compression strength. The experimental results are to be analyzed for establishing national standards, and International Standards (ISO Technical Committee 206).

This project has not yet been approved by the VAMAS steering committee because it has proven difficult to secure a broad international participation in this project. Interested participants have been identified in Japan and in the US, however the testing capabilities needed are quite specialized and several laboratories would have problems conducting the tests. Discussions are ongoing as to how to secure additional participation.

*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 to NPL.

A New Work Item proposal for Mode II is to be launched in 2002, together with consideration given to publishing a TTA on this topic. Dissemination of the fibre fragmentation work for interface strength measurements is also expected in the coming year. The VAMAS fatigue coordinator, Dr G D Sims, has been asked to finalize the English text for the ISO 13003 standard on cyclic loading of composite materials, that incorporates results/procedures from prior VAMAS work.

Project 1, Assessment and Recommendations to ISO on Mode II Test Methods, has been completed. The project was organized 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 the “4-point ENF” procedure is the preferred method for standardization. No further progress has been made since ISO TC61 accepted the preferred method for the Mode II standard. A NWI full ballot will be proposed. In addition, publication of a TTA is being actively considered.

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.

The project leader retired at the end of August, but remains active part time through a guest scientist position in the Building and Fire Research Laboratory at NIST. Although this limits his ability to do more experimental work in the VAMAS program, it does give more time to analyse the data and write up the results. Six laboratories have completed the tests and returned their results. A number of characterization experiments have been performed on the model system, both the resin and the fibre. The experimental work is completed and the final report is expected in 2003. Publication of a TTA is being actively considered as the best method of disseminating the full results. A full ISO NWI ballot is unlikely to be successful at this point in time, particularly if financial support is required to directly support the these final stages of the standardisation process. The major route for dissemination of results is publications and presentations at scientific meetings. In addition, the detailed information in the database will be distributed to anyone who is interested in studying the single fibre fragmentation test and its data analysis. The participants at Michigan State gave a preliminary overview of the results at the American Society for Composite Meeting in Purdue University, September 2002. There was in the proceedings a paper from that meeting which will be an initial dissemination vehicle, but this paper will not contain details or all of the conclusions. The project leader hopes to present a full paper at an appropriate international meeting in the next 2 years

Project 3, Measurement of Through-thickness Testing Properties, continues to be delayed. 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.

Work has not started, as the EU Framework 5 network proposal to support the VAMAS project submitted in 2000 was unsuccessful and the project leader changed posts and is unable to lead this item without financial support. A UK or USA leader will be sought. NPL has a new project (started1/4/2001) on round-robin validation of the drafts previously prepared for through-thickness tension, compression and shear test methods, and therefore may be able to take on the leadership of this project. Dissemination is planned to be via International conferences (e.g. ICCM/12, CTS/5, ECCM-9), standards committees (ISO TC61/SC13/WG16 and CEN TC 249/SC2/WG5 as draft methods and/or precision data in support of such drafts) and via EU Framework 5 network.

Project 4, Assessment of Damage Tolerance for Polymer Matrix Composites, is being reformulated following a change in project leadership to NPL. The delayed damage tolerance project may be encouraged by a Japanese proposal to fund AFNOR for the “compression-after-impact” test that falls within this topic. NPL will be involved as successful with a recent bid for a three year project on “defect criticality and is interested in leading this project.

New Proposals

Other topics under consideration include:

- Thermal analysis techniques (DSC, DMA), based on enquiries received from other countries (e.g. Italy, USA) to join a recent UK Studio Project.

- Structural element tests, such as open hole compression and tension, and pin bearing tests. These tests cover more than just aerospace interests. Some intercomparison of NPL proposals and ASTM preferences, as well as the wider generation of precision data to supplement a UK round-robin.

- Ultrasonics for: a. defect detection during NDE inspection,
  b. measurement of elastic constants.

- Composite adhesion tests (peel-static/dynamic, weathering, edge delamination (fatigue), heat cycle) related to:
  a. composite bonded repair of structures, or
  b. composite stiffened conventional materials.
  (Proposed under IEA/OECD Climate Initiative (CTI) by Japan).

- Collaboration on design data database formats based on the technical data sheet standard, ISO 10350-2, drafted at NPL.

- Collaborative international long-term durability studies to generate a larger database.

- Processing properties test methods.

A survey of national co-ordinators is underway to determine priorities for future programmes. There is a need to define programmes attracting real support. Composites is maturing as an industry and as product codes are produced, needing their own support (e.g. design codes), missing infrastructure aspects will be highlighted.

*Technical Working Area 13*

## LOW CYCLE FATIGUE

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The 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 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 as VAMAS Report No. 41. A meeting of TWA13 National Representatives was held during the 6th Int. Conf. on Biaxial/Multiaxial Fatigue & Fracture, Lisbon, Portugal, June 26-29, 2001.

The chairman attended the ASTM fall meeting, Dallas, Texas, November 2001, where an informal agreement has been reached to exchange information between this TWA, ISO/TC164/SC5 and ASTM E08.05.01 groups. It was also agreed to exchange all draft and final reports and other relevant documentations and outputs. As part of revising ASTM E606 standard on low cycle fatigue testing, ASTM intends to conduct an inter-laboratory test programme. An invitation was received from the ASTM to NPL and the other members of TWA13 to take part in this exercise.

Work has started within ISO/TC164/SC5/WG11 towards developing a standard for alignment measurement in uniaxial fatigue testing machines, which will be based on the recent VAMAS-EC joint project. Whilst the chairman left NPL, 31 January 2002, it has been agreed in principle that he will continue his role within VAMAS until 30 September 2002 when the current activities of this TWA are due to be completed.

# METAL MATRIX COMPOSITES

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The focus of the TWA is to develop an understanding of the mechanical and physical property determination of metal matrix composites with heterogeneous microstructures at room temperature and elevated temperatures, and to establish reliable test methods through a series of intercomparison exercises.

There are 4 materials available for examination but only 1) and 2) are currently being studied:
1) SiCw/A2009 whisker reinforced MMC, supplied by NASA
2) SiCf/Ti-15-3 continuous fibre reinforced MMC (from Textron), supplied by NRIM
3) SiCf/Al continuous fibre reinforced MMC (from 3M), supplied by NRIM
4) SiCp/Al particulate reinforced MMC, supplied by NPL

1) SiCw / A2009 MMC

The initial focus of project 1, tensile and fatigue test methods for silicon carbide whisker reinforced A2009 MMCs at room and elevated temperatures, was directed at a room temperature procedure. This resulted in 1997 in the preparation of ISO/TTA 2 (Technology Trend Assessment), tensile test discontinuously reinforced MMC at ambient temperature. Recent results from tensile test method at elevated temperature will be included in the ISO TTA document and for consideration as an annex on modulus measurement to the current ISO EN 1000-2 standard for tensile testing. There was a meeting of the CEN committee on 13th February in Luxembourg 2002.

(2) SiC fibre /Ti-15-3 MMC

The initial efforts in project 2, tensile and fatigue test methods for continuous fiber reinforced MMCs at room and elevated temperatures, focused on the role of specimen size during fatigue testing. The fatigue exercise will be started once the questionnaire returns have been received. It is hoped that between 6-10 organizations will participate.

Proposed Future Projects:

A meeting of TWA 15 was held at National Physical Laboratory, London on 27 March 2002. A new questionnaire has been prepared and this was distributed to TWA 15 national representatives, participants and contacts database. A number of areas for future work on MMC have been discussed, and suggestions for new work areas included fracture toughness, creep, thermal fatigue testing, physical properties, microstructural characterization, damage assessment and life prediction methods and interfacial mechanical property tests. The next meeting of the TWA will be held in 2003 at the International Conference on Composite Materials (ICCM-14) on 14-18 July 2003 in San Diego, California USA.

Standardization Activities and Dissemination

Contact with ASTM continues with representation on the ASTM D30 committee on MMC, and NPL is also actively involved in a European Thematic Network on MMC - "MMC ASSESS", co-ordinating the effort on test methods and mechanical properties. A dedicated web site has been set up on the VAMAS TWA 15 activity with a link to the VAMAS web site, which will provide further opportunities for the dissemination of the TWA 15 and VAMAS activities.

*Technical Working Area 16*

## SUPERCONDUCTING MATERIALS

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There are currently seven projects underway, including two initiated this past year.

In Project No. WG1-1, 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, after bending each of the specimens to a different, designated curvature at room temperature. A round-robin has been implemented with 12 participant laboratories. Three kinds of Bi-2223 superconducting tape samples and a set of bending devices were delivered to each of participants. The coefficient of variation (COV, the standard deviation divided by the average) was very small for the bending strain of 0.2%, increased at 0.4% strain, and remained at the same level of 6% up to 1.0% strain. A part of the data scatter came from the inhomogeneity in strain dependence of critical current among specimens while the rest came from the insufficient specifications of test conditions. It was concluded that measurements should be done under more specified and strain-controlled conditions. Studies to further clarify the origins of the data scatter are still underway among some of the participants for the establishment of the standard method.

The objective of Project No. WG1-2, 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. It was concluded that the resistive method should be recommended as the standard method. An IEC standard draft for this critical temperature measurement method was proposed as a committee draft at the IEC/TC90 meeting in 2000. Modifications have been made on the scope, definition and measurement details. An international standard is expected to be published in 2002.

The objective of the new Project No. WG1-3, Measurement Method of Irreversibility in Oxide Superconductors, is to clarify the relation between V-I characteristics and flux pinning, determine the relation among irreversibility (critical) fields obtained by different methods, and establish an adequate and reliable measurement method. The project was initiated in 2001. In Kyushu Inst. Tech., the irreversibility fields of a typical Bi-2223/Ag tape were measured by a variety of methods and it was demonstrated that the irreversibility fields obtained can be plotted against the electric field criterion, converted from different criteria, on a unique curve. This implies that all the methods examined are acceptable for the measurement of irreversibility field as far as the criterion is well defined. The four-probe method is, however, recommended as the standard technique, since the method is the easiest and the most reliable method among all.

In Project No. WG1-4, Coupling Loss Measurement in Multifilamentary HTS Superconductors, the objective is to examine the adaptability of the VSM method to measure the coupling loss of multifilamentary HTS Superconducting tapes. The project was initiated in 2001. A round-robin test was proposed in which the ac loss of LTS and HTS tapes is measured as a function of field ramp rate up to 4 T/min. The project leader and TWA officer are inviting participants and looking for samples appropriate for the test.

In Project No. WG2-1, 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. The trapped flux density profile of a bulk is measured by scanning a Hall probe on the surface of the specimen. The magnetization process in which a strong field is applied to a specimen during cool-down performs the flux trapping. For trapped-field measurements, the trapped flux density is strongly affected by the geometry of the specimens, which is known as the de-magnetizing effect in conventional permanent magnets. It has been clarified that such effect can be corrected by a simple calculation method. The correction curve was made for standardization.

Recently, the trapped field profile was found to be sensitive to the active size of magnetic field sensor, density of sampling points and the gap distance between the magnetic field sensor and superconductor. It was also found that the trapped field itself was strongly related to the shape and aspect ratio of the sample. Now the bulk group is preparing a round-robin test, in which the trapped field mapping is done at liquid nitrogen temperature for selected samples. The findings obtained by the round-robin test will be fed into the IEC standard draft currently under discussion.

In Project No. WG3-1, Measurement Methods for the Surface Resistance in Thin Film Superconductors, the objective is to confirm that the target accuracy should be 20% or less for the measurement method adopted in IEC draft standard, IEC 61788-7 on the surface resistance in thin film superconductors. The surface resistance is determined by applying a microwave signal to a cylindrical dielectric resonator sandwiched between two superconducting thin films and measuring the insertion attenuation of the resonator at a certain frequency between 8 GHz to 30 GHz. The third round-robin test (3 Japanese and 1 United States) was implemented in August 2001, in which a unique pair of sapphire rods was used to minimize the error from dielectric inhomogeneity. The resulting data scatter was less than 10 % and small enough. The 4th round-robin test (domestic) was implemented in March 2002, in which each institute used their own pair of sapphire rods. The resulting data scatter was also about 10 % and small enough.

The IEC standard draft, IEC 61788-7 is now at the IS stage, published January 2002

The objective of Project No. WG4-1, 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. The measurement is performed at room temperature using a mechanical test machine. The first round-robin test was implemented in 2000 among 8 participant laboratories. In this round-robin test, the measurement conditions were not specified at all as all participants were requested to measure using their own techniques. For all three Bi-2223 superconducting tape samples, the data scatter (COV) of the 0.2% proof strength and the tensile strength was rather small among laboratories, while that of the Young modulus and elongation was large. The reasons for the large scatter are now under discussion. The next round-robin test will be implemented in a near future, where the measurement conditions are well specified.

*Technical Working Area 17*

## CRYOGENIC STRUCTURAL MATERIALS

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A draft for ISO/TC164/SC1 "Metallic Materials - Tensile Testing in Liquid Helium" has been approved as a Committee Draft at the last SC1 meeting held in Turin in September 2001. The Committee Draft Document ISO/CD 19819 is being balloted as a draft international standard.

Currently, there are three projects underway. Project No. 5, 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. The results of the third round robin with the advanced specimen geometry were good as expected and preparation of a Technology Trend Assessment (TTA) document for ISO/TC 61 is underway.

The objective of Project No. 6, 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. The results of round-robin test on titanium alloy have been analyzed and evaluated. The magnetic effect on the value of Young's modulus was not observed in the proposed testing system. A TTA document for ISO/TC164 is in preparation.

Project No. 7, 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. The results of the round-robin test were analyzed and evaluated with the result that the JETT and CT tests gave almost the same fracture toughness. A TTA document for ISO/TC164 is in preparation.

Proposed/Future Projects:

1. Young's modulus measurement during tensile testing at liquid helium temperature
2. Tensile tests for composite material at cryogenic temperatures.
3. Promote programs on basic testing to increase number of participants from industry.

## STATISTICAL TECHNIQUES FOR INTERLABORATORY STUDIES

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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 important to secure the necessary close contact of statisticians with the scientists responsible in the field of application. Members of this TWA or statisticians from the coordinating organization should be involved in new projects from the initiation of the project. During the past year, TWA 18 has reviewed new project proposals from TWA 2 and TWA 3.

In general it is not intended that the TWA initiates its own projects. Objectives of the TWA are:

- to provide statistical and computational support in the phases of the design and analysis of classical interlaboratory studies on the basis of existing guidelines, standards and computer programs (Service),

- to provide mathematical, statistical and computational support for optimizing specimens, processes, experiments, interlaboratory studies and test methods by approaches of experimental design and quality engineering (Taguchi), and mathematical modelling and computer simulation (Optimization),

- to identify needs and undertake corresponding problem-oriented research and development on statistical models and computational approaches for collaborative projects of selected structure and purpose (Research) and

- to offer consultation and help in mathematics, computational statistics, scientific computing, selected fields of chemometrics, and material science and engineering (Consulting).

TWA 18 can additionally offer statistical advice for problems that cannot be addressed on site and also may give other TWAs assistance finding a source for statistical consulting if it is not available in-house.

## MEASUREMENT OF RESIDUAL STRESS

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Currently TWA 20 is concerned with the measurement of residual stress by neutron diffraction. Testing and analysis of results on this project have been completed. Emphasis is now on developing a standard and on disseminating the findings to potential users. Presentations of the procedure have been made to a seminar hosted by ORNL, Oak Ridge, USA in October 2001 and to a residual stress network meeting at UMIST, Manchester, UK in February 2002. One of the members (Dr. T.M. Holden) of the TWA visited Australia in February 2002 to give advice to ANSTO.

An ISO/VAMAS Technology Trends Assessment document has now been issued as a proposed procedure for the measurement of residual stress by neutron diffraction. CEN/TC 138 AHG 7 is now using this document as a basis for the preparation of an international standard in association with ISO/TC 135 with representation from national standards organizations.

The results of the investigation have been distributed widely. A web site has been set-up to report the findings. Its address is; http://www.risoe.dk/vamas-twa-20/. Presentations of the results were given at ICRS-6, Oxford, UK from 10-12 July 2000 and at THERMEC 2000, Las Vegas, USA from 3-8 December 2000.

Publications for the period are as follows;

WEBSTER, G.A., 'Development of a standard for the measurement of residual stress by neutron diffraction', Proceedings ICRS-6, 10-12 July 2000, Oxford, Institute of Materials, ISBN I-86125-123-8, Vol. 1, 189-196.

WEBSTER, G.A & WIMPORY, R.C, 'Non-destructive measurement of residual stress by neutron diffraction', THERMEC 2000, Las Vegas, USA, Dec 2000.

*Technical Working Area 21*

# MECHANICAL MEASUREMENTS FOR HARDMETALS

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A revised ISO standard, ISO 3327, Bend Strength Tests for Hardmetals based on a previous TWA21 project has now been completed. It is being used extensively by a world-leading hardmetal company to support market penetration of very high strength round products.

The current ongoing project is aimed at developing good practice in the toughness testing of hardmetals. Nine laboratories from 5 countries are participating with corresponding support from 5 other laboratories in 3 more countries. Ten batches of material from 4 organisations were supplied for the evaluation of four test methods, including two geometries of the short rod/bar test, the Palmqvist test, and a notched beam test. The test outline was reported in VAMAS Bulletin No. 24, 2001. The project has been slow to start due to the need to prepare an extensive number of polished samples for Palmqvist toughness tests. The first sets of data have been returned from participants and it is expected that there will be sufficient data to begin a preliminary analysis by midsummer 2002. This will be followed by consideration by partners before a final report is issued in early 2003. Finally, documents for consideration by the standards community will be prepared in spring 2003.

Regular progress reports are given to

- EHMG, European Hard Materials Group (part of EPMA, European Powder Metallurgy Association) – meets every 6 months.
- BHARG, Research Group of British Hardmetal Association – meets every 3 months. Members also include VAMAS participants from USA and Germany.
- Recipients of NPL Hard Materials Newsletter – database of > 400 worldwide.
- ISO TC119/SC4 on Cemented Carbides/Hardmetals.

Future Project
A possible project has been identified based on the confusion between customers and suppliers in Europe and North America where specifications may require different hardness test methods. A comprehensive exercise to evaluate the underpinning reasons for the differences between Rockwell (depth sensing) and Vickers (indentation diameter) is likely to be well supported. This has been discussed at a recent ISO meeting in the UK in November 2001. Other possible project areas could include technical support for a standard on grain size measurements.

*Technical Working Area 22*

# MECHANICAL PROPERTY MEASUREMENTS OF THIN FILMS AND COATINGS

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Chairmanship of TWA 22 has passed from Stuart Saunders at NPL to Douglas Smith, formerly a Vice-Chairman, at NIST. Nigel Jennett continues NPL's strong leadership role within the TWA, as Vice-Chairman. Vice-Chairman Toyonobu Yoshida has recently notified the other TWA 22 officials that he would like to step down as Vice-Chairman, and has recommended Professor Osamu Takai, of the University of Nagoya, as his replacement. This nomination is supported by all TWA 22 officials, and the approval of Professor Takai as Vice-Chairman by the Steering Committee will be requested.

The objective of Project 1, Measurement of Hardness and Young's Modulus of Thin Coatings Using Depth Sensing Indentation Instruments, is the development of methodologies for the determination of the properties of a coating *in situ*, and validation of the method by carrying out an international round-robin. This project is now essentially complete, except for the writing of the report. Round robin data have been received from all participants. In spite of the large number of participants, an ambitious test matrix resulted in a relatively low number of responses for some tasks, making it difficult in those cases to reach statistically significant conclusions about the results. Nevertheless, a very detailed draft report has been prepared by the project leaders at NPL. It is expected that the few final issues regarding the presentation of the results will be settled within the next few months, and the report will then be available for distribution.

The objective of Project 2, Adhesion of Thin Coatings, is to evaluate and compare several test methods for adhesion of thin coatings ($<10\ \mu$m), including bend testing and indentation, through round robin testing, with the long-term goal of developing a simple, quantitative engineering test for coating adhesion. Work items on adhesion measurement have been agreed to by CEN TC 184 WG5, and the progress to be made in this project will directly input into the CEN activity.

Much of the discussion at last year's reporting meeting in San Diego (ICMCTF01, May 2001) focused on the types of adhesion tests that should be emphasized in a round robin effort, and the types of materials that should be used. As in previous years, a broad consensus was not reached, but a significant number of the participants favored the use of common, commercially available tests. It was also felt that at least three tests should be compared, using specimens with at least three levels of interfacial strength (for the same film and substrate materials).

Such sets of specimens have now been prepared by NPL. They consist of CrN films, several micrometers thick, on stainless steel substrates. Interfacial strength is controlled by variation of a Pd/Au interlayer. The steel coupons are of a size (100 mm x 10 mm x 3 mm) that will allow a wide variety of common adhesion tests to be performed on them, including scratch testing, indentation and various tests involving a ductile deformation of the substrate (i.e. 4-point bend and uniaxial tensile strain). We are now in the process of lining up round robin participants willing to test the specimen sets.

In addition to the CrN specimens, several Japanese laboratories have studied a new set of TiN coatings on thin stainless steel substrates. Led by Dr. Takahito Omura, this group looked at delamination of the coatings resulting from large tensile strain in the substrates. Difficulties were encountered, however, as many of the steel substrate foils failed before reaching sufficient strain to cause coating delamination.

The objective of new Project 3, Elastic Properties of Thin Films and Coatings led by Dr. Uwe Beck, BAM, is the determination, evaluation, and inter-comparison of elastic properties of layered systems derived from (i) localized destructive Instrumented Indentation Testing (IIT), and (ii) non-localized non-destructive Surface Acoustic Wave (SAW) spectroscopy measurements.

Although this project has only just been approved, several oxide films have been prepared and are being characterized by BAM. The films should be available for preliminary tests by NIST and AIST within the next month or two.

NEW PROJECTS

None are formally proposed, but it is generally agreed that a project on residual stress measurement for films and coatings should be proposed.

*Technical Working Area 24*

# PERFORMANCE RELATED PROPERTIES FOR ELECTROCERAMICS


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Dissemination and awareness of TWA 24 continues through our external collaborations and through conference presentations, notably the National Measurement Congress, UK in November 2001, the IEEE standards subcommittee on Loss in acoustic materials (piezoelectrics), and CENELEC BTTF63-2 (Advanced Technical Ceramics), a task force of the CENELEC Technical Board responsible for standard BTTF63-2, including WG 1 (Methods of Test for Ceramic Electrical Insulating Materials and Components) and WG 2 (Methods of Test for Piezoelectric and Electro-Optic Ceramics). The Chairman of TWA 24 is actively involved in the EC Thematic Network POLECER, Polar Electroceramics, with separate tasks focused on standardization issues. Major standards-based outcomes from this network will be channeled through TWA 24. The UK Piezoclub held a meeting on 24 October 2001 where our VAMAS Project Number 1 - Direct method for piezoelectric coefficient - was described. The TWA24 home pages are sited within the Functional Materials Group Website at NPL: http://www.npl.co.uk/npl/cmmt/functional/vamas.html

There have been some developments in the CENELEC BTTF63-2 standards subcommittee. The standards documents that describe the measurement methods and descriptions for multilayer actuators are in final draft stage. Very recently, some serious issues have arisen regarding the funding of future BTTF63-2 WG2 activity, which the convener is investigating. A thick-film piezoelectric standards document may also be written in the future, depending on securing sufficient EU funding. TWA 24 directly supports and has influence over these standards committees.

Project 1 – International Intercomparison of Direct Piezoelectric Coefficient using the Berlincourt Method.

Presently, standards that exist demonstrate the appropriate methods for measuring the material's linear properties. However, the use of piezoelectric ceramic materials as sensors and actuators is increasingly being hindered by the material's inherent non-linear properties. As the materials are being used at higher stresses, electrical fields and temperatures, then these non-linear and lossy properties become more important and new methods are needed to fully characterize them. Piezoelectric ceramics produce a charge when mechanically stressed and this measurement has been adopted as an industrial standard known as the Berlincourt Method. In this test, a small static preload is applied to a ceramic disc and an even smaller ac cyclic load is then superimposed. The resultant charge is measured across the faces of the (poled and electroded) ceramic test piece.

The Berlincourt method has enjoyed wide use and acceptance by industrial users and producers of piezoelectric materials for quality control of ceramic pieces and materials data generation for design. The method has been extensively studied by the NPL Materials Centre and shown to be most sensitive to the manner in which the experiment is conducted. To test the variability between different laboratories in measuring this parameter (d33) is to test the robustness of the measurement method. The data generated would help determine the real level of scatter in the measurement of the direct piezoelectric coefficient via this method.

The project is in the final stages of completion. A recent report written for the POLECER network presents the current progress made. POLECER aims to promote the application of polar ceramic materials and to educate manufacturers and users in their use. Central to this goal lies the role of standards and measurement good practice.

In the first round robin, results have been so far received from 11 laboratories (industrial and academic). The ceramic discs were initially measured by NPL using the measurement good practice developed during a UK DTI co-funded project involving 3 UK companies. Many sets of samples were posted out, all measured initially at NPL and chosen with almost identical values. Participants were asked to measure the value of d33 using the method that they would normally adopt (all the methods were based on the Berlincourt technique). Four ceramic discs were supplied to each participant, one soft composition and one hard composition of two different thickness, and they were asked to measure each sample 10 times to determine an average value. There are a number of interesting points to describe:

a. Variation in a batch:

The 10 mm samples generally had a lower standard deviation than that of the 1 mm samples. This is potentially due to the increased variation of mechanical stress within each 1 mm sample compared with the 10 mm samples because of the loading method (point contact in most cases). This non-linearity of $d_{33}$ with stress has been described in the NPL Good Practice Guide No.33. The individual tests carried out by each laboratory seem consistent, evidenced from the small error bars. This is a measure of the repeatability of the test method.

b. Variation between batches - between laboratories. Importance of test set-up/method:

It is instructive to explore the variation in the data outliers. In one laboratory, only one measurement per sample was recorded. However, a very large preload and ac cyclic load was used. One 10 mm sample and two 1 mm samples showed lower values than laboratory average value. This may be expected from the d33 variation with ac load and preload noted by NPL in earlier work. The outlier from another laboratory was also measured at very high ac cyclic stresses that may explain this large difference from both samples.

c. General Comments:
Variation between laboratories was larger than that recorded within the batch indicating the importance of experimental method. Additionally, a larger variation between laboratories was found for the 1 mm (thinner) samples than for the 10 mm samples. This makes sense based on the different stresses present within the 1 mm compared with the 10 mm samples. [See non-linear $d_{33}$ with stress in GPG No.33]. So, factors that affect $d_{33}$ (Berlincourt) include stress state via preload, ac load, and indenter profile. There is a larger variation in the soft 1 mm samples than the corresponding hard materials, demonstrating their increased sensitivity with stress state. A large variation between laboratories may be due to lack of awareness of the importance of preload, ac load and indenter type. These are not currently controlled or recorded. This round robin has identified these issues and others reported in the final report and may be the subject of our follow-on interlaboratory exercise.

Project 2 - Measuring $d_{33}$ from Displacement Dependence of Electric Field Strength.

Single crystal piezoelectric materials actuate to significantly higher values of strain for the same electrical field level compared with conventional polycrystalline piezoelectric ceramics. These new materials are in need of standardization support so that their development may be suitably and robustly compared world-wide. The assessment of the electrical field dependence of the piezoelectric coefficient may be carried out by recording the displacement under high electrical field for one complete electric field cyclic loop. The material's non-linear properties may then be evaluated. This project will compare results using an International Interlaboratory exercise and will use polycrystalline materials and the new single crystal piezoelectric materials.

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

| | |
|---|---|
| Project 3 | Measurement of piezoelectric strain at high electrical/mechanical stress |
| Project 4 | Measurement of piezoelectric and dielectric properties at high stress |
| Project 5 | Measurement of electrical and mechanical fatigue of piezoelectric ceramics materials |
| Project 6 | Properties of electrical conductive, optical transparent thin films |
| Project 7 | Thermal effects on performance |
| Project 8 | The dielectric, elastic and piezoelectric properties (matrix elements) need to be 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 9 | The temperature dependence of the complex coefficients is important in some applications (such as high power and space applications). |
| Project 10 | Hydrostatic property measurements to about 14 MPa are important for underwater applications. |
| Project 11 | The meaning of fatigue / aging / degradation to be clarified, particularly the distinction between reversible and irreversible mechanical or electrical damage |
| Project 12 | In project 1 it would be interesting to introduce measurements of piezoelectric coefficient of thin and thick films on substrates. |

*Technical Working Area 25*

## CREEP/FATIGUE CRACK GROWTH IN COMPONENTS

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The aims of TWA 25 are to:

- Establish accurate and reliable procedures for assessing creep/fatigue crack growth at elevated temperatures in components, which contain defects.
- Determine procedures for analysing the test data using fracture mechanics concepts.
- Validate results against measurements on standard laboratory specimens using the ASTM E1457-98 procedure and the new BS 7910 assessment procedure.
- Outline recommendations in procedures for dealing with component creep/fatigue crack growth testing and analysis.

The details of the meeting minutes, future meetings and additional information about participating is contained on the VAMAS TWA 25 website at http://mesm.org

*Previous and Current Work Tasks*

| **Title** | **Start/End Date** | **Output** |
|---|---|---|
| Collect shared information on component tests | Nov 99 – Nov 00 | Database - collected |
| Round Robin review of industrial test methods | Jan 02 | Review Document – completed and disseminated |
| Round Robin crack growth prediction on a Pipe & a Plate | Dec 01 – Dec 02 | Document – report of different analysis methods to be prepared |
| Establish appropriate list of specimen geometries and testing and analysis methodology | Jun 02 – Feb 03 | Document – annex in a draft Code of Practice |
| Publication of a special issue in the Int. J. of Pressure Vessel and Piping | Jun 02 – Mar 03 | Journal publication of the partners findings |
| Draft Code of Practice for Creep/fatigue testing of components | Jun 02 – Dec. 03 | Document – Draft Code of Practice |

### Other Planned Output and Dissemination Activities in the Next 2 Years

- Meet together with experts from industry and research institutes in order to disseminate information, seek their continued collaboration and complete the tasks set out above.
- Collaborate with API, ASME and ASTM E08 Creep fracture committee to share information.
- Collaborate with 'CRETE' FP6 EEC funded project on 'Creep crack growth testing of industrial specimens' to disseminate and share information.
- Disseminate findings in various relevant conferences and meetings.
- Preparing the draft Code of Practice to establish reliable methods for prediction of component behaviour.

### Benefits of Participation to Competitiveness of Industry

Creep and creep/fatigue crack growth properties measured on laboratory specimens can be applied to real components. Industry will benefit from an improved lifetime assessment methodology that a standardized component testing and analysis method will produce. Over 30 universities, institutes and industrial firms from around the world have actively participated in this project in the first two years. It is expected that this interest will continue. There has therefore been a useful flow of information to the project by those associated with it. UK industry, especially the power generation sector, will benefit as a result of this project both due to the final deliverable of the Code of Practice and also due to the fact that UK's present methodology on creep crack growth will be placed to the forefront and is likely to be adopted by international organizations such and CEN, ASME, API and ASTM.

*Technical Working Area 26*

## FULL FIELD OPTICAL STRESS AND STRAIN MEASUREMENT

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A TWA 26 meeting was held at the SEM conference in Portland, Oregon, USA on Tuesday 5th June 2001. The agenda for the meeting was as follows:

| | |
|---|---|
| Introductions | Dr Richard Burguete<br>Prof Eann Patterson |
| ASTM E08 - Standard guide for evaluating non-contacting optical strain measurement | Prof Mike Sutton |
| Activities on standardization of full-field optical stress and strain measurement methods in Japan | Prof. Yoshihari Morimoto<br>Satoru Yoneyama |
| Inspection of Nuclear Plant using Optical Methods | Dr Yoshihiro Yamashita |
| The SPOTS initiative in Europe | Prof. Eann Patterson |
| New projects | Dr Richard Burguete |

<u>Extracts from the ASTM E08 minutes</u>
In 1998 Mike Sutton was asked by Dr. George Lucas (formerly chairperson for an NDE subdivision of ASTM and a manager at MTS Corporation) to give a talk at the E28 subcommittee on calibration issues for non-contacting methods. A small task force was set up (E08.03.03), with Peter McKeighan as co-chair, to draft a Standard

Guide. This was completed in the fall, 1999 using existing ASTM format as a guide. The revised document was presented to the subcommittee at the November, 1999 meeting. After modifications, it was approved for submission as a full ASTM ballot at the May, 2000 meeting.

The Draft Standard Guide was balloted in the summer/fall of 2000 and apparently approved without negative ballots, eventually being sent out for publishing as an ASTM Standard Guide in the spring, 2001. Unfortunately, it was learnt in April, 2001, that one negative ballot had been received. The goal is to re-ballot the standard in the fall of 2001. Once this standard guide is in place, it allows everyone to compare methods based on the same terminology, providing a solid basis for determining which methods are best suited for a specific application.

Standardization activities in Japan

Currently standards exist for strain gauge methods, but no standards exist for full-field optical methods. The activities of TWA 26 were presented at JSNDI 32nd Symposium on stress-strain measurement and strength evaluation (Jan 2001) and at JSME-MMD 2001 Spring symposium (March 2001). A full session was held at the JSEM annual conference (28-29 June 2001) covering the present situation in Japan with regard to the standardization of full-field optical methods. The Japan Society for Photoelasticity was renamed Japan Society for Experimental Mechanics (JSEM) and the Standardization of Full-field Optical Stress and Strain Measurement division has been established within the JSEM under the chairmanship of Prof Morimoto.

Results of a questionnaire on optical stress and strain measurement method use was presented. Data was gathered at JSME-MMD and through the JSME website. There is currently little interest in standards in many Japanese companies. The Japan division of TWA26 will continue to disseminate the activities of TWA 26 and promote the importance of standardization in Japan.

Life extension of Japanese nuclear power plants from 40 to 60 years has required more rigorous and sensitive inspection regime. Laser methods are being developed and used for flaw detection and sizing. The importance of reliable and verifiable data is obvious and standardization is the main way to realize this.

The SPOTS initiative in Europe

An Expression of Interest was submitted by Eann Patterson to the EU in response to the 5th Dedicated call (Call identifier: GROW /DC5MTI). This was successful and lead to a dedicated call under ‘I. MEASUREMENT AND TESTING; (i) Methodologies to support standardization and Community policies; Pre-normative topics: Optical techniques for strain measurement (topic V.15)’. A consortium of 10 submitted a proposal (co-ordinated by Eann Patterson), the results of which will be announced soon. It must also be assumed that the success of the EOI has in part resulted from the registration to CEN/STAR for prioritization of Co-normative and Pre-normative research.

Proposed/Future projects

The following project proposals are being drafted and are being discussed. The first was due for ratification in May 2002 by the group that will be carrying out the work. The proposal for the second project is currently being prepared.

1. The ASTM Standard guide for evaluation of non-contacting optical strain measurement systems. A project initiation form has been drafted and was to be discussed at he next meeting of task group E08.03.03. This meeting was held in Pittsburgh on 6th May 2002. Dr Peter McKeighan agreed to add this to the agenda for the meeting and a country co-ordinator for the USA would be appointed if he were forthcoming. It is intended that a more formal link between this ASTM group and VAMAS is established.

2. Round robin on i) displacement and strain calibration for industrially based ESPI systems and ii) effect of system parameters on fringe processing.

*Technical Working Area 27*

# CHARACTERIZATION METHODS FOR CERAMIC POWDERS AND GREEN BODIES

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This Technical Working Area was approved in May 2000 and the kick-off meeting held November 9, 2000 at Sandia Labs, in Albuquerque, New Mexico. The objective of this TWA is to develop and standardize 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

The first project initiated, "Determination of coarse particles in advanced ceramic powders," led by Dr. Cho, Chonnam National University, South Korea, was designed to refine the testing procedure through a preliminary round robin before starting the round robin on a wider international scale. The international round robin was carried out by six participants: University of Nagaoka, Japan; JFCC, Nagoya, Japan; BAM, Germany; Asahi Glass Company, Yokohama, Japan; NPL, UK; & University of Chonnam, Korea.

The results of this preliminary test have shown that the testing procedure has to be corrected with regard to a general applicability of the method to commercial powders and suspensions. This has been done in a second preliminary round robin of which the results were discussed during a project meeting at the PacRim 4 conference in Maui, Hawaii and in Washington, DC, on March 19, 2002. As a result of this meeting the international final round robin was started on April 4, 2002. The results of this round-robin will be used to improve the ISO TC 206 PWI 03.

A potential new project, "Determination of the isoelectric point of diluted ceramic suspensions by the microelectrophoresis method" is in the planning stage.

*Technical Working Area 28*

# QUANTITATIVE MASS SPECTROMETRY OF SYNTHETIC POLYMERS

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The first meeting of the TWA was held in conjunction with the Chicago meeting of the American Society of Mass Spectrometry (ASMS). Current members include:

Dr. Charles M. Guttman, NIST Polymers Division, USA
Professor Iondela, University of Alabama-Birmingham, USA
Dr. Christina B. Jasieczek, PPG Industries, USA
Dr. Shinichi Kinugasa, NIMC, Japan
Professor Liang Li, University of Alberta, Canada
Professor Jimmy Mays, University of Tennessee, USA
Dr. Maurice Montaudo, University of Catania, Italy
Dr. Philip Price, Union Carbide Corp., USA
Dr. Steffen Weidner, BAM, Germany

Current project:
1. Method Development for MALDI TOF MS of Low Molecular Mass Polystyrene

Three members of the VAMAS TWA 28 met at the Chicago ASMS Meeting. It was decided to write a preliminary Guide (or Practice) on the use of MALDI for the molecular mass distribution (MMD) determination of Polystyrene (PS) of molecular masses from 1000 u to 70000 u. The general outline of the guide would follow that of the DIN or ASTM method for the GPC determination of the MMD of PS and be based on the protocol used in the recent Interlaboratory comparison of PS by MALDI MS organized by NIST and BAM. PS was chosen because it was successful run at 8000 u in this interlaboratory comparison. TWA 28 members present thought it should be the first polymer for which standard test methodology should be developed since PS is best controlled in a MALDI situation. The Chairman developed a preliminary draft of the guide for PS. The draft was sent to all members of the TWA for comments and suggestions about areas needing research. Comments were received and a second revision is in process.

Three members of the committee have begun discussions with various national and societal standards organizations for inclusion of our results in their standards. ASTM D20 on Plastics in the United States has initiated a task group on MALDI MS of synthetic polymers that will be led by Charles Guttman.

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