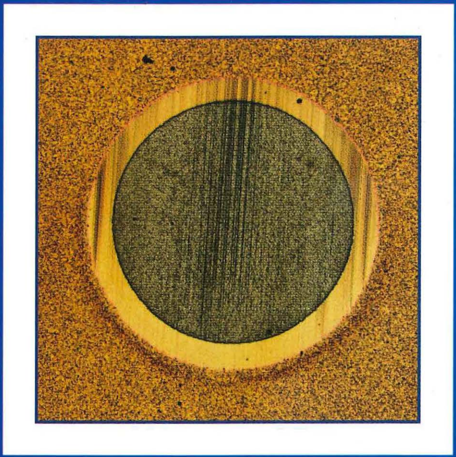
VAMAS



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April 2005

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: Typical crater produced in a ball cratering wear test with 2% SiC abrasive

Photograph courtesy of:
Mark Gee, Engineering and Process Control Division, NPL, UK

VAMAS

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• Chairman's Message •

I am honoured to report to the VAMAS community during my short tenure as Chairman of the VAMAS Steering Committee, prior to passing the Secretariat to NIST on behalf of the USA.

Firstly, can I thank Colin Lea, the past Chairman, for his work on behalf of VAMAS over many years. Colin has retired from the NPL Materials Centre, but is continuing in a Corporate Role. I have been involved for the last ten years in VAMAS through TWA 5 Polymer Composites, firstly as a project leader for fatigue test methods, and latterly as TWA Chairman. My role at NPL in the Division of Engineering and Process Control includes science responsibilities for the materials work alongside the national measurement programmes on mass, length and temperature including the related SI units. This is an appropriate association in view of the VAMAS interactions with BIPM (Bureau International des Poids et Mesures - www.bipm.fr), the home of the Metre Convention and SI units.

Secondly, I would like to provide an update on this important initiative, which has been discussed at recent meetings of the Steering Committee. The initiative concerns materials metrology; firstly its definition (see below) and secondly, identifying the underpinning techniques or "tools" (e.g. mechanical testing for modulus, thermal analysis techniques) and their traceability requirements to either SI units or reference data or images.

> Materials Metrology
> "Development and application of traceable measurements and predictive modelling to the structure, property and performance of materials throughout the life cycle enabling their efficient use."

The use of modern materials pervades all of industry and has a strong influence on its competitiveness. The world market for materials-related products has increased rapidly in recent decades and is more than 10^{11} US dollars a year. Nevertheless, there has been less effort devoted to establishing a worldwide equivalence of materials properties and reference materials, which are required to reduce technical barriers to trade in materials products, than to researching new materials.

During the last six months discussions have been held with BIPM regarding formalising the metrology content of materials research and standardisation. On behalf of VAMAS, I am preparing the case for establishing a Working Group on Materials Metrology to further investigate the need for this work. We are interested in suggestions from you, for work areas that are both important in materials measurement and similar in nature to the key comparisons undertaken in other areas of metrology by Consultative Committees (CCs), or involving interaction with existing CCs on materials aspects.

It should be noted that the Korean National Metrology Institute, KRISS, representing the APEC (Asia-Pacific Economic Community) countries has been involved in these discussions as part of the Steering Committee's wish to widen the number of countries involved in directing VAMAS work. Already 120 participants from non-G7 countries have contributed to the technical work of VAMAS, alongside over 600 G7 participants. This globalisation will be a major feature of the continuing work of the VAMAS Secretariat.

Finally, I would like to thank all participants in VAMAS, the Steering Committee and the TWA chairmen – who form the dynamic hubs of our activities. Both Colin and myself would like to record our appreciation of the effective work undertaken by Martin Rides, as Secretary, for the last three years and to wish the NIST team, Stephen Hsu, Chairman (previously TWA 26 chairman) and Stephanie Hooker, Secretary our best wishes for the next period as custodians of VAMAS at a very influential time.

Graham D Sims
Chairman

Contact details for 2005-2008

Chairman: Dr Stephen Hsu
National Institute of Standards and Technology
Room A-263, Building 223
100 Bureau Drive, Gaithersburg, MD 20899
USA
Tel. 00 1 301 975 6120
E-mail: stephen.hsu@nist.gov

Secretary: Dr Stephanie Hooker
NIST - Materials Reliability Division
325 Broadway, Boulder, CO 80305
USA
Tel: 00 1 303-497- 4326
E-mail: shooker@boulder.nist.gov

Past Chairman and BIPM initiative:
Dr Graham D. Sims
National Physical Laboratory,
Teddington, Middlesex, TW11 0LW, UK
Tel: 00 44 20 8943 6564
E-mail: graham.sims@npl.co.uk

• Feature Article •

VAMAS TWA 20 Measurement of Residual Stress
Case Study

Prof. G. Webster
Imperial College of Science, Technology and Medicine
London, SW7 2BX, UK
Tel: +44 20 7594 7080
Fax: +44 20 7594 7017
e-mail: g.webster@ic.ac.uk

Project 01 in this Technical Working Area (TWA) is concerned with the development of reliable procedures for the non-destructive measurement of residual stress in materials and components by neutron diffraction. An ISO/VAMAS Technology Trends Assessment document (TTA 3) has been issued describing these procedures. This document is currently being considered by CEN TC/138 AHG 7 (in association with ISO TC 135) for issue as a draft standard with the provisional number prCEN ISO TS 21432.

Weldments are a major source of residual stress in components. Usually fracture in welds initiates from sites of high residual tension. In most components the magnitude of any residual stresses present is not known. As a result design codes and safety assessment procedures make recommendations about the residual stresses that should be allowed for in welds for safe operation. To be conservative these stresses are usually substantially greater than may be actually present. Consequently considerable benefit can be gained if the recommended stresses can be reduced.

A study has been carried out using the procedures described in TTA 3 to measure the residual stresses generated in 3 sizes of T-butt weld (shown in Fig 1) of a low carbon ferritic steel. The measurements were made through the toe of the weld (Fig 2) along YY where fracture is most likely to occur. The transverse stresses (i.e. those in direction x perpendicular to YY) along YY for the 25mm thick weld, obtained at a number of neutron sources, are illustrated in Fig 3. Broad agreement is obtained between the different sets of results. Similar data were obtained for each size of weld. It can be seen, as shown in Fig 4, that the results can be normalized with respect to plate thickness so that they can be expected to be relevant to a range of sizes of geometrically similar T-butt welds.

Suggested average and upper bound residual stress distributions (normalized with respect to yield stress) that can be employed for all the T-butt welds are indicated in Fig 5. A comparison between the upper bound, other data and recommendations provided in a British Standard code is given in Fig 6. It is evident that the code provides a substantial overestimate and that significant benefit (in terms of greater operating stresses or extended lifetimes) can be gained from use of the measured upper bound distribution. This distribution is currently being considered for inclusion in the code.

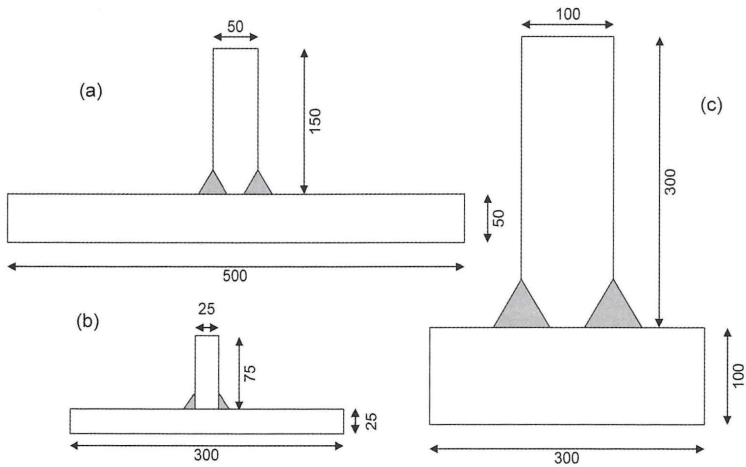

Figure 1. Cross sections of T-plate weld samples used in this investigation (all dimensions in mm)

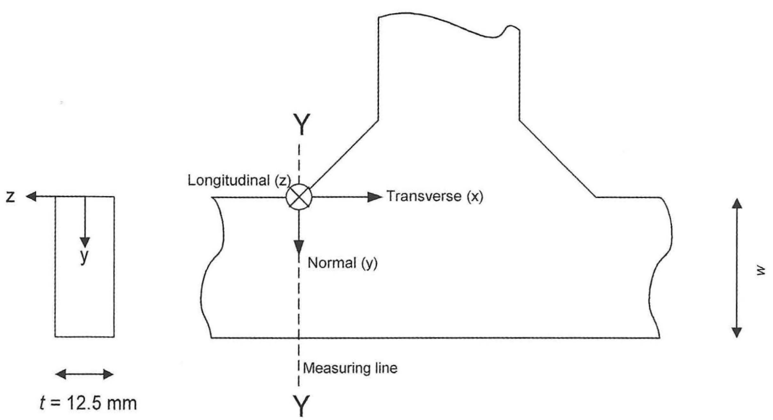

Figure 2. T-plate weld with measurement directions indicated, where the origin of the axes is at the weld toe.

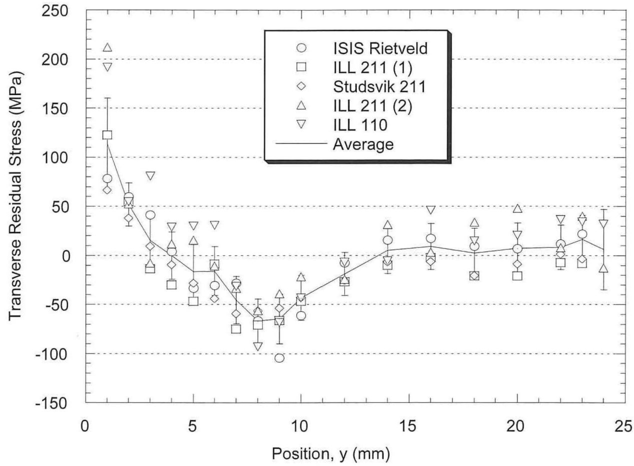

Figure 3. **Individual data sets of 25 mm T-plate weld residual stresses in transverse direction**

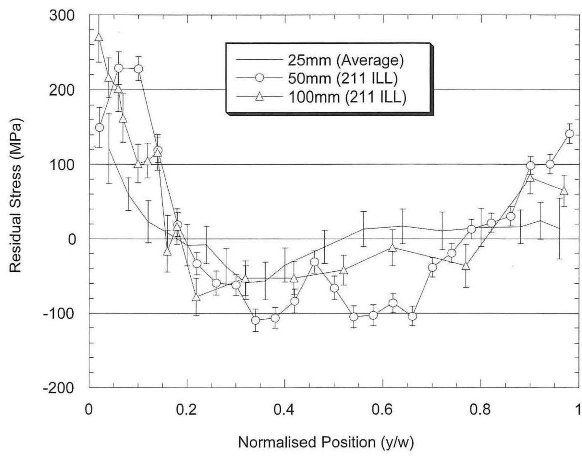

Figure 4. **Comparison of measured transverse stress distributions in all welds**

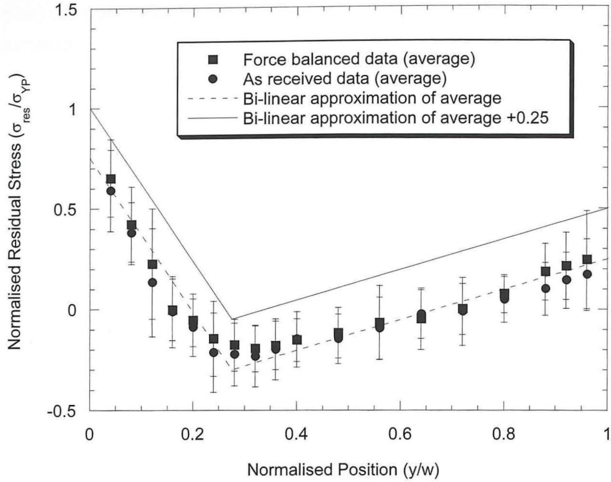

Figure 5. **Normalized transverse residual stress distributions showing bi-linear estimations of average and upper bound of data from Fig 4.**

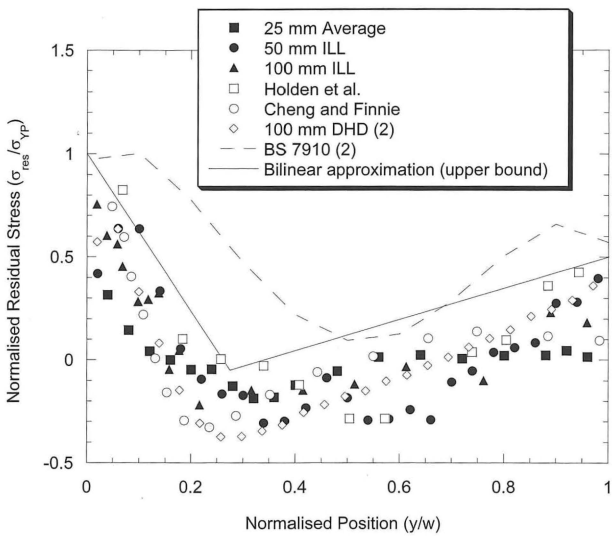

Figure 6. **Normalized upper bound transverse residual stress distribution compared with other experimental data and BS 7910 recommendations.**

• Feature Article •

VAMAS TWA 13 on Low Cycle Fatigue: An Overview of its Work and Achievements

Dr. F. A. Kandil
EuroTest Solutions Limited
PO Box 1226, Kingston, UK, KT2 5YJ
Tel: +44 20 8541 5244
Fax: +44 20 8541 5520
E-mail: fathy.kandil@eurotest-solutions.co.uk

1. Introduction

Low cycle fatigue (LCF) tests simulating in-service conditions are known to be difficult to devise and perform, expensive to execute and can frequently give unreliable results. They, however, remain essential for the safe design and operation of components occasionally stressed into the plastic region.

VAMAS Technical Working Area (TWA) 13 was created in 1987 to investigate the above under the chairmanship of Dr David Gould of the CEC. The author became chairman in 1995.

2. VAMAS First Inter-laboratory Study on LCF testing

The objective of this study [1] was to generate sets of elevated temperature LCF data representative of accepted testing practices at the time and, subsequently, to identify those aspects of the testing procedure that significantly affect the repeatability and reproducibility of the results. The ultimate aim was to provide the underpinning knowledge for defining a valid standard test procedure.

This study, initiated by the European Commission and extended via VAMAS, involved 16 European and 10 Japanese laboratories. In view of the potential influence of material characteristics on LCF life, the programme involved the testing of four materials classified according to their cyclic strain characteristics - AISI 316L at 550 ^{o}C (strain hardening), 9Cr1Mo at 550 ^{o}C and IN 718 at 550 ^{o}C (strain softening) and Nimonic 101 at 850 ^{o}C (strain stable). The test conditions, including strain ranges, strain rate, wave-shape, temperature and limits to specimen surface finish, were specified in the guidelines circulated to the participants. However, each laboratory used its normal test methods, i.e. specimen form and size, specimen manufacture, extensometry, test machine and recording equipment. Twenty-one data sets were supplied for AISI 316L steel, 16 for 9Cr1Mo steel, 12 for IN718 and 12 for Nimonic 101.

Overall it was shown [1] that for all materials, the within-laboratory variation in lifetimes was within a factor (N_{fmax}/N_{fmin}, where N_f is the number of cycles to failure) of two. This was significantly less than the between-laboratories variation, which was up to a factor of approximately sixty. The variability increased as the total strain range to which the specimens were subjected decreased. It was noted that the spread in the IN718 data was the greatest, followed by that for the Nimonic 101 alloy and 9Cr1Mo whilst AISI 316L steel showed the least inter-laboratory variability. The use of different definitions of failure had little effect on the spread of the data. Consideration of sub-sets of the data showed that limiting the analysis to data from smooth specimens reduced the spread but only marginally because of the generally shorter lifetimes of ridged specimens. Figure 1 shows an example of inter-laboratory results for 9Cr1Mo at 525 °C.

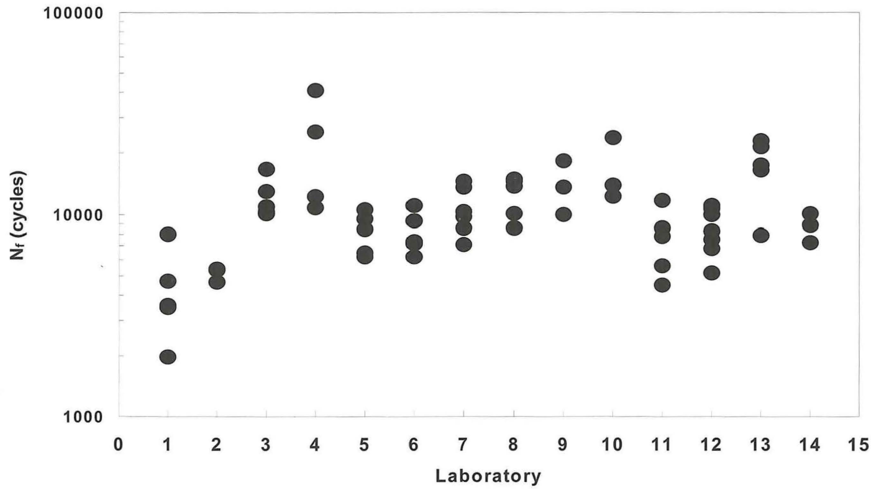

Figure 1. Inter-laboratory results of fatigue life for 9Cr1Mo steel at 525 °C (strain-controlled tests, $\Delta\varepsilon_t$ =0.6%)

Further analyses concluded [2-5] that the primary causes of inter-laboratory data variability were most likely associated with specimen bending due to misalignment in the test system's load-train, lateral rigidity of the test system, and errors in measuring and controlling the strain and the test temperature.

The findings from this study led to the production of a new procedure for measuring specimen bending in LCF testing [6].

3. VAMAS Second Inter-laboratory Study on LCF testing

The main objectives of the second inter-laboratory study [7-9] were to (i) produce a framework for quantifying the measurement uncertainties in LCF testing and (ii) reduce the variability of between-laboratories lifetime data to within a factor of five.

Some forty LCF tests at ambient temperature were carried out by five laboratories on specimens that were machined in one laboratory and made of the same batch of Nimonic 101 superalloy material. Special care was taken to align all the test facilities and the aim was to reduce the maximum bending strain due to misalignment of the test system to below, whichever criterion is applicable, 50 micro strain or 5% of the applied elastic axial strain. The fatigue life test results from these tests showed remarkable agreement [see Fig. 2] between laboratories so that the variability in lifetime was less than a factor of two.

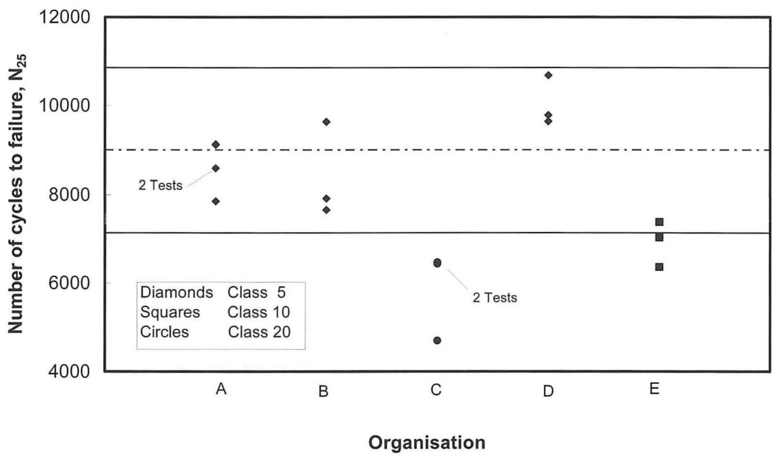

Figure 2. Inter-laboratory results of fatigue life, N_{25}, for Nimonic 101 at ambient temperature (strain-controlled tests, $\Delta\varepsilon_t$ =1.0%). Also shown are the estimated uncertainty limits based on the data for laboratories A, B & D who had Class 5 machine alignment [7].

A further 30 LCF tests were performed at 850 °C. Only three laboratories were involved in this part and each made their own specimens from the same batch of Nimonic 101 superalloy material. The resultant variability in lifetimes was within a factor of six.

In addition, eighteen LCF tests at 850 °C were carried out by one laboratory on one machine with predetermined levels of specimen bending. These tests showed that as the specimen bending (which was determined at an applied average axial strain of nominally 1000 micro strain) increased from approximately 2% to 40%, fatigue life for tests with $\Delta\varepsilon_t$ =1.1% decreased by approximately one half.

Although the number of participants in the recent series of round robins was significantly lower than in the previous study, the recent results indicated an order of magnitude improvement on the earlier results. This was achieved by adhering to newly developed measurement practices.

One major impact of the above EC/VAMAS studies has been to increase awareness amongst LCF testing practitioners of the importance to properly measure and reduce specimen bending due to misalignment in the load-train in LCF test systems. Almost all major LCF test machine manufacturers now produce commercially so-called “alignment fixtures” and associated software that enable fine adjustments of the alignment of the test machine’s load-train to be made. More information on the main outputs from this study can be found in [e.g. 7-9].

4. Contributions to standards

An explicit objective of VAMAS is the transfer of its pre-standards research to standards organisations and the community at large. Early coupling of pre-standards work with actual standards development has long been identified as the preferred and most efficient route to an approved and recognised standard.

Active coupling of this work with standards development activities was, in effect, built in from the start of this work. VAMAS TWA 13 has institutional links and representatives in all the interested organisations in the standardisation process in this field today (ISO, ASTM, CEN, BSI and JSI). To this were added government and other industrial forums with the end result being an international working group on LCF that interacts and works directly with both industry and standards organisations. Several standards already published or currently being developed are either wholly based on or partly benefit from the outcome of the EC/VAMAS TWA 13 studies. These include:

- ISO/TC164/SC5/WG11/NP 12XXX (new work item) – Metallic materials – Fatigue testing – Axial alignment of fatigue machines.

- ISO 12106:2003, Metallic materials – Fatigue testing – Axial strain-controlled method.

- ISO/NP 12111, Metallic materials – Fatigue testing – Thermal-mechanical method.

- ASTM E606 (revision under development), Standard Practice for Strain-Controlled Fatigue Testing.

- ASTM E1012 (revision under development), Standard Practice for Verification of Specimen Alignment under Tensile Loading.

- CEN Workshop 11 – Part One: Uncertainty evaluation in LCF testing.

- BS 7270 (revision under development), Method for Constant Amplitude Strain Controlled Uniaxial Fatigue Testing.

5. Conclusions

The VAMAS work has:

- identified ways in which the variability in inter-laboratory low-cycle fatigue life data can be reduced to a factor of less than 5.

- produced a new procedure for verifying the alignment of uniaxial test machines.

- contributed to the development, so far, of seven standards on fatigue testing and test machine alignment.

6. Acknowledgements

The studies reported here were partially funded by the participating organisations and by the Commission of European Community through the Standards, Measurement and Testing programme.

The author gratefully acknowledges the financial contribution made by NPL towards the production of this article.

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Technical Working Area 1

WEAR TEST METHODS

Dr. E. Santner
Bundesanstalt für Materialforschung und-prüfung (BAM)
D12200 Berlin, Germany
Tel: +49 30 8104 1810
Fax: +49 30 8104 1817
e-mail: erich.santner@bam.de

Status of Recent Projects

The objective of Project No. 1, Compilation of Wear Test Standards, was to provide a validated source for standardized wear test methods that can be used by engineers and tribologists. The project has been completed and a CD with the database of standardized methods is available (see *http://midas.npl.co.uk/midas/WTSindex.jsp*).

The objective of Project No. 2, Ball Cratering Wear Testing, is to perform pre-standardization work in order to establish the "Ball cratering wear testing" method for the abrasion testing of coatings as a standard and to produce a recommended procedure for carrying out the test.

In the test a rotating ball is pressed against a test sample in the presence of a fine abrasive slurry. This creates a spherical depression on the surface of the sample that can be measured to determine wear. Two different types of test can be performed. These are the perforating test, where the wear rate of both the coating and substrate can be derived from measurement of the dimensions of craters that penetrate through the coating, and a non-perforating test where the coating is not penetrated.

The test method was validated through an interlaboratory exercise that had 14 participants from Europe, Japan and the USA. The draft procedure has been written as a draft CEN and a draft ASTM Standard.

Funding was gained from the EU (Project CRATER) to support the standardization of this test method. This project is now drawing to a close with the completion of the interlaboratory exercise that has been carried out.

Technical Working Area 2

SURFACE CHEMICAL ANALYSIS

Dr. C. Powell
Surface and Microanalysis Science Division
National Institute of Standards and Technology
Gaithersburg, Maryland, USA 20899-8370
Tel: +1 301 975 2534
Fax: +1 301 216 1134
e-mail: cedric.powell@nist.gov

Overview

Surface chemical analysis is used extensively for a wide range of purposes in science and technology. Technologically, surface and interface properties are crucial for the fabrication and performance of a wide range of advanced materials (e.g., ceramics, composites, alloys, polymers, superconductors, diamond-like thin films, and biomaterials), semiconductor devices, optoelectronic materials, high-density magnetic-storage media, sensors, thin films, and coatings. Surface or interface integrity is critical for many properties or processes, such as the electrical behavior of a semiconductor device, the wear or corrosion of an automobile part, or the degradation of an implant material in the human body. Surface analysis is used in these applications for failure analysis in manufacturing or of a component in its service environment as well as for monitoring steps in product fabrication or process development. The surface composition may be required for product quality control or may be usefully correlated with specific material properties, processes, or phenomena (e.g., corrosion, adhesion, lubrication, wear, segregation of bulk impurities to interfaces, and diffusion, amongst many others) so that improvements can be developed. Surface and interface properties are also critical in many areas of public concern ranging from new sources of energy (e.g., photovoltaics and fuel cells) to defence systems (e.g., sensors and stability of high-power laser optical components) and health and environmental problems (e.g., particulate pollutants in the atmosphere and the stability of stored nuclear waste). The most commonly used surface-analysis techniques are Auger-electron spectroscopy (AES), secondary-ion mass spectrometry (SIMS), and X-ray photoelectron spectroscopy (XPS). These techniques are utilized in different modes to obtain information on the near-surface composition and, in conjunction with ion sputtering or other methods to remove surface layers, to determine the composition of the material as a function of depth from the original surface. For many advanced materials (e.g., semiconductor devices, magnetic-storage media, and new classes of nanomaterials), the materials are fabricated with critical dimensions on the nanometer scale and there is little distinction between surface, bulk, thin-film, and interface properties. A growing need in these applications is to determine composition as a function of position, particularly in the vicinity of surfaces and interfaces, and to measure critical dimensions of thin-film systems and nanostructures.

Three new TWA 2 projects were initiated during the past year (Projects A8, A9, and A10). Project A8 will evaluate new methods for the determination of lateral resolution of surface-analysis instruments using a novel multilayer stack of different III-V semiconductors being developed at the Federal Institute for Materials Research and Testing (BAM) in Berlin. The exposed surface of the stack has a strip pattern with strip widths ranging from about 0.3 nm to 700 nm, and it is expected that this material will be useful for determining the lateral resolution of scanning Auger microscopes and other surface-analysis instruments. Project A9 will evaluate procedures for automated peak detection in XPS. It is planned to conduct an interlaboratory comparison using simulated spectra with test peaks of varying shape and amplitude to test the effectiveness of different peak-detection algorithms and available peak-detection software. Project A10 will assess the accuracy and precision of thickness, density, and roughness measurements of thin-film structures determined by grazing-incidence X-ray reflectance (XRR) in an interlaboratory comparison. This project is believed to be timely in view of the growing use of XRR for characterizing thin-film materials and the recently completed pilot study of measurements of the thicknesses of 1.5 nm to 8 nm SiO_2 films on silicon by M. P. Seah *et al.* [Surf. Interface Anal. 36, 1269-1303 (2004)]. This pilot study was conducted under the auspices of the Consultative Committee on Amount of Substance and involved thickness measurements by SIMS, XPS, XRR, and seven other methods.

Many of the outputs of TWA 2 projects have been incorporated or are in the process of being incorporated into ISO standards. Table 1 shows the titles of standards or draft standards of ISO Technical Committee 201 on Surface Chemical Analysis arising from or related to TWA 2 projects (ISO: International Standard; TR: Technical Report; WD: Working Draft; CD: Committee Draft; DIS: Draft International Standard). TWA 2 is a category-A liaison body with ISO/TC 201 and eight of its nine 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, convenors of ISO/TC 201 working groups, or as experts on ISO/TC 201 working groups.

Table 1. Titles of ISO/TC 201 standards documents arising from or related to TWA 2 projects (with the corresponding TWA 2 project number shown in parentheses).

ISO/TC 201 subcommittee	Title of standard, draft standard, or technical report
SC2: General Procedures	WD 16268: Ion-implanted surface-analytical reference materials—Procedure for standardizing the retained areic dose in a working reference material (5)
SC3: Data	ISO 14975: Surface chemical analysis—Information formats

Management and Treatment	(10,29,30) ISO 14976: Surface chemical analysis—Data transfer format (10) DIS 22048: Surface chemical analysis – Information format – Static secondary ion mass spectrometry (10,29,30) WD 22474: Surface chemical analysis – Auger electron spectroscopy and X-ray photoelectron spectroscopy – Guide to methods for detecting peaks (A9)
SC4: Depth Profiling	ISO 14606: Surface chemical analysis—Sputter depth profiling—Optimization using layered systems as reference materials (1) TR 15969: Surface chemical analysis—Depth profiling—Measurement of sputtered depth (1,5)
SC5: Auger Electron Spectroscopy	ISO 18118: Surface chemical analysis—Auger electron spectroscopy and X-ray photoelectron spectroscopy—Guide to the use of experimental relative sensitivity factors for the quantitative analysis of homogeneous materials (17,20) CD 18392: Surface chemical analysis – X-ray photoelectron spectroscopy – Procedures for determining backgrounds (A6) CD 18516: Surface chemical analysis – Auger electron spectroscopy and X-ray photoelectron spectroscopy – Determination of lateral resolution (A8) ISO 19318: Surface chemical analysis—X-ray photoelectron spectroscopy—Reporting of methods used for charge control and charge correction (A2) TR 19319: Surface chemical analysis – Auger electron spectroscopy and X-ray photoelectron spectroscopy – Determination of lateral resolution, analysis area, and sample area viewed by the analyser (A8) CD 20903: Surface chemical analysis – Auger electron spectroscopy and X-ray photoelectron spectroscopy – Determination of peak intensities (A6)
SC7: X-ray Photoelectron Spectroscopy	ISO 15470: Surface chemical analysis—X-ray photoelectron spectroscopy – Description of selected instrumental performance parameters (A8) ISO 15471: Surface chemical analysis—Auger electron spectroscopy – Description of selected instrumental performance parameters (A8) ISO 15472: Surface chemical analysis—X-ray photoelectron spectroscopy—Calibration of energy scales (2) ISO 17973: Surface chemical analysis—Medium resolution Auger electron spectrometers—Calibration of energy scales for elemental analysis (2)

	ISO 17974: Surface chemical analysis—High resolution Auger electron spectrometers—Calibration of energy scales for elemental and chemical state analysis (2) WD 18327: Surface chemical analysis—X-ray photoelectron spectroscopy—Guidelines for estimating unintended degradation in a material (A5) ISO 21270: Surface chemical analysis – X-ray photoelectron and Auger electron spectrometers – Linearity of intensity scale (9,23) DIS 24236: Surface chemical analysis – Auger electron spectroscopy – Repeatability and constancy of intensity scale (9) DIS 24237: Surface chemical analysis-X-ray photoelectron spectroscopy – Repeatability and constancy of intensity scale (23)
SC8: Glow Discharge Spectroscopy	ISO 14707: Surface chemical analysis – Glow discharge optical emission spectroscopy – Introduction to use (A4)

The next meeting of TWA 2 national representatives, project leaders and observers will be held in Vienna, Austria in association with the 11th European Conference on Applications of Surface and Interface Analysis to be held on September 25-30, 2005.

Current projects

The objectives of the current projects are presented below:

Project 13: Tests of Algorithms for Data Processing in AES – Factor Analysis

The project's objective is to develop a reference factor analysis procedure that gives spectral intensities, spectra of unknown components, number of components and allows the identification of contributing factors in the spectral mixture. The factors that influence the accuracy of the procedure should be characterized.

Project 14(d): Tests of algorithms for angle-resolved XPS

The project's objectives are to establish the validity and accuracy of the ARXPS technique, and to determine:

- the amount of substance within the outermost 0-10 nm of a solid; and
- the in-depth composition profile within the outermost 0-10 nm of a solid

It is planned to use an approach similar to that used in Project 14 (c) to determine the accuracy of XPS peak shape analysis for determination of nano-structures by analysis of the background signal of inelastically scattered electrons. Comparisons will be made of results obtained from ARXPS with results obtained by the XPS peak shape analysis technique evaluated in project 14 (c) as well as by RBS, XRF, PIXE and SIMS.

Project A3(b): Time-of-flight SIMS interlaboratory study - Evaluating repeatability and reproducibility and testing G-SIMS

The main objective is to study the repeatability and reproducibility of Time-of-Flight static SIMS studies on conductors and insulators. A subsidiary objective is to evaluate the effectiveness of "gentle" SIMS (G-SIMS) in various laboratories.

Project A3 has been subdivided into three closely related projects dealing with the repeatability and reproducibility of static SIMS measurements. Project A3(a), "Static SIMS Interlaboratory Study – A Survey of the Issues," was completed in 2000. This work led to Project A3(b), "Time-of-Flight SIMS Interlaboratory Study – Evaluating Repeatability and Reproducibility and Testing G-SIMS," that was initiated in 2001 and will be completed in 2005. A third phase of the work, "Static SIMS Interlaboratory Study – Repeatability and Constancy of the Relative Intensity Scale, Calibration and Quantification," is planned to start in 2006 as Project A3(c).

Project A6: Evaluation of uncertainties in XPS peak intensities associated with different techniques and procedures for background subtraction

The project's objectives are: (1) to determine the magnitudes of uncertainties of XPS peak intensities arising from commonly used techniques and procedures for spectral background subtraction; and (2) to make recommendations for improved methods of background subtraction that will lead to improved accuracy in quantitative surface analyses by XPS.

Project A7: Evaluation of electron beam damage of SiO_2/Si in Auger microprobe analysis

While many investigations have been made of electron-irradiation damage of silicon dioxide during analyses by Auger electron spectroscopy (AES), it is very complicated or tedious to determine the critical dose for such damage from intensity changes of the silicon L_3VV Auger spectrum versus irradiation time. Instead, it is proposed to measure the decomposition cross section (DCS) of a SiO_2/Si sample due to electron irradiation from intensity measurements of the metallic Si L_3VV peak. The critical dose for damage of SiO_2 can then be calculated from the DCS values of SiO_2 and SiO. It is proposed to conduct an interlaboratory study to evaluate the effectiveness of this proposed method. If this study is successful, it will establish the method as a means of investigating electron-beam-induced damage of oxides during AES analyses.

Project A8: New procedures for the determination of lateral resolution of instruments for surface analysis in the nanometre range

Evaluation of new methods for the determination of lateral resolution of instruments for surface analysis.

Project A9: Evaluation of Procedures for Automated Peak Detection in X-ray Photoelectron Spectra

X-ray Photoelectron Spectroscopy (XPS) spectra must be analyzed to detect peaks prior to elemental/chemical assignment. ISO/TC201/SC3/WG4 has identified three algorithms that appear to be useful for peak detection in XPS. It is planned to prepare simulated XPS spectra with weak peaks of varying shape and amplitude to evaluate the effectiveness of the algorithms for peak detection identified in the draft ISO document and of similar algorithms available in commercial software or incorporated in the software of commercial XPS instruments.

Project A10: X-ray reflectivity measurements for evaluation of thin films and multilayers

The objectives are to: (1) Establish accurate and reliable procedures for making non-destructive X-ray reflectivity (XRR) measurements. (2) Choose test samples, grown with different density and thickness, and examine by transmission electron microscopy (TEM) and X-ray reflectivity to determine thickness and density, respectively. (3) Conduct interlaboratory comparison to establish reproducibility and errors. (4) Assemble the necessary information for preparing a draft standard for making XRR measurements

CERAMICS FOR STRUCTURAL APPLICATIONS

Dr. K. Breder
Saint-Gobain Abrasives
Higgins Grinding Technology Center
1 New Bond Street, MS 413-201
Worcester, MA, USA 01605-0008
Tel: +1 508 795 4147
Fax: +1 508 795 4283
e-mail: Kristin.Breder@Saint-Gobain.com

Currently there are two projects in the working area. In both projects only the final report is still pending.

Determination of Phase Composition and Percent Crystallinity in Hydroxyapatite (TWA 3 Project 14)

Hydroxyapatite (HA) is a ceramic material increasingly used as a biocompatible monolithic material or a coating encouraging acceptance of an implant by 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 goal is, therefore, to ascertain whether the proposed method is suitable for standardisation by ISO to enable characterisation of materials to the specification of ISO 13779-1 and ISO 13779-2.

From an initial group of 23 participants, results were obtained from a total of 9 participants from 6 countries. The results from the round robin are briefly summarized below:

- The reproducibility of the method for powder specimens is acceptable,

but

- Accuracy of the method is unacceptable.

In order to verify the low reproducibility for analysis of plasma sprayed specimens additional analysis of several specimens was performed by the organiser. The work was discussed at the ISO meeting in Vancouver Canada, September 04. The project was completed in 2004. The final report is pending.

Inert Flexural Strength of Alumina (TWA 3 Project 16)

The objective of this project is to determine the need for assessing the inert strength of alumina in a specific inert environment, and subsequently the need to put such specifications into the existing standards for measuring flexural strength of advanced ceramics.

The project is a joint round-robin between VAMAS and APEC Network for Materials Evaluation Technology (ANMET), a new organization established to promote cooperation in materials evaluation technology for the economic benefit of the Asia-Pacific region. A total of 14 participants in 10 countries participated in this round robin, and data has been received from all participants. The results show that the inert environments of flowing nitrogen and paraffin oil were equally suited for determination of inert strength, while the testing in ambient air consistently resulted in lower flexural strength. The data also showed a relatively large spread in results between laboratories, and it was also obvious that the dataset contained low strength outliers. Additional statistical analysis has been performed and will be included in the final report.

Coordination with ISO TC 206, Fine Ceramics is still good at the current B liaison level, and the coordination with ASTM C-28 Ceramics is also very good (the TWA chair is also the vice-chair of ASTM C-28).

Proposed Future Projects

This TWA group has traditionally worked on projects related to mechanical properties of monolithic ceramics, and the results from these have had significant impact on numerous international standards. Current work in ASTM and ISO is moving towards applications related standards, and less work related to properties testing and development of properties methods seem to be ongoing in the various laboratories that have been active in this area. Discussions have been held regarding round robins related to strength testing by diametral compression (Brazilian Disc) and biaxial flexure, and also about the influence of grinding conditions on flexural strength. At the present time there are no proposed projects related to any of these ideas. The needs and interests, including specific needs from ISO, and collaboration under the MOU with the IEA will be further assessed.

Future Meetings

A working group meeting is planned in January 2005 in conjunction with **The 30th Annual Cocoa Beach Conference** to be held in Cocoa Beach Florida, USA January 2006.

POLYMER COMPOSITES

Dr. G. Sims
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex, UK, TW11 0LW
Tel: + 44 20 8943 6564
Fax: + 44 20 8614 0433
e-mail: graham.sims@npl.co.uk

There are presently two completed projects for dissemination, a third awaiting input from the ISO committee and a new project provisionally approved on multi-axial testing.

The standard ISO 13003 on cyclic loading of composite materials, that incorporates results and procedures from the VAMAS fatigue project, has now been published.

Status of current projects:

Project 1: Assessment and Recommendation to ISO on Mode II Test Method

The objective was to assess several competing Mode II test methods in order to make a recommendation to ISO, thereby over-coming a veto on progression of Mode I.

The project led by Dr. Davies (IFREMER, France) identified the four-point end notched flexure (4ENF) as the preferred method and precision data was obtained for two material systems supplied by Japanese and USA companies. An ISO NWI full ballot for Mode II will be proposed. In addition, publication of a TTA is being actively considered. The European group, ESIS, is planning to provide the required documentation.

Project 2: Measurement of mechanical properties for the fibre-matrix interface

The program 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. The work was led by Dr. Hunston (NIST, USA) and involved 7 researchers from 5 countries. The fibre used in the study was donated by Hexcel.

The results were presented at the ECCM – Brugge meeting. The participants at Michigan State gave a preliminary overview of the results at the American Society for Composites Meeting in Purdue University. Publication of a TTA is considered as the best method of disseminating the full results.

Publications on this work include:-

M J Loderio, "Investigation of PMC interface properties using the single-fibre fragmentation technique", NPL Report MATC (A) 16, 2001.
D A Mendels, "Analysis of the single-fibre fragmentation test" NPL Report MATC (A) 17, 2001.
M J Loderio, "Single-fibre fragmentation test for the characterisation of interfacial phenomena in PMCs", NPL Measurement Note MATC (MN) 07, 2001.

Project 3: Assessment of Damage Tolerance For PMCs

The objectives of this project are to determine a philosophy for damage assessment in composite materials by;

- assessing available techniques
- developing new techniques and procedures
- validating test methods developed
- obtaining precision data
- generating a database of damage tolerance test data

The delayed damage tolerance project may be enabled by a Japanese proposal to fund AFNOR for the "compression-after-impact" test that falls within this topic. This proposal has now successfully passed ballot and been added to the ISO work programme. Development of the ISO standard will take account of a similar ASTM document in preparation. VAMAS is awaiting identification of specific aspects requiring study and validation in the experimental round-robin

NEW PROPOSAL: Multiaxial Testing

A new project "Measurement of Multiaxial Properties of Polymer Composites" is being developed. There are several methods of creating multiaxial loads, including use of axial forces and pressure (internal/external) using tube specimens, biaxial plate or cruciform type configurations and full rig systems applying combinations of axial/bending/twisting loads. The biaxially loaded cruciform specimen has been identified as of most interest currently. The objective of this project is to propose acceptable methodologies that will lead to internationally accepted test methods for the evaluation of multiaxial strength of fibre reinforced composites. It is envisaged that these will form the basis of a new international standard.

This project has been approved by the Steering Committee providing there is world-wide involvement. Participation is initially from Belgium and UK. The proposal is led by QinetiQ (UK). Further participants should contact the TWA Chairman as above.

Technical Working Area 13

LOW CYCLE FATIGUE

Dr F A Kandil
EuroTest Solutions Limited
PO Box 1226, Kingston, UK, KT2 5YJ
Tel: +44 20 8541 5244
Fax: +44 20 8541 5520
e-mail: fathy.kandil@eurotest-solutions.co.uk

Overview

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 Reports No. 41. Recent Intercomparisons on Low Cycle Fatigue and Alignment Measurements and No. 42. A Procedure for the Measurement of Machine Alignment in Axial Testing. An article "*VAMAS Technical Working Area 13 on Low Cycle Fatigue: An Overview of its Work and Achievements*" is presented as a feature item in this Bulletin.

TWA 13 has continued contributing to the developments of the following international and UK standards:

ISO/TC164/SC5/WG11 – Measurement of machine alignment.
A new version was produced and discussed in Beijing, China, 12-14 October 2004. It was agreed to split the standard into two parts, one dealing with the verification of machine alignment and the other dealing with the measurement of specimen bending. At the next meeting, which will be held at NPL in October 2005, the updated draft will be submitted as NWIP ballot.

CEN Workshop 11 – Part One: Uncertainty evaluation in LCF testing.
The final version is being prepared for publication in 2005.

ASTM E1012 – Measurement of specimen bending due to non-alignment
A new version was balloted in February 2005.

BS 7270 – Strain-controlled LCF testing.
The final version is being prepared for publication in 2005.

The TWA chairman gave a presentation titled "*Uncertainty in Fatigue Test Data: Similarities and Differences Between ASTM and CEN Approaches*" at the ASTM E08.05 sub-committee meeting in Washington DC, November 2004. An article based on this presentation is being prepared and will be published in due course.

The TWA 13 chairman contributed to the following international standards meetings:

- CEN Workshop 11, "Measurement Uncertainty in Mechanical Testing", in Brussels in June 2004
- ISO/TC164/SC5 meeting in Beijing, China, in October 2004
- ASTM E8 and E28 meetings in Salt Lake City, UT, May 2004 and in Washington DC, November 2004

Future relevant meetings

- 10-14 October 2005 – ISO/TC164/SC5, "Fatigue Testing", NPL, UK
- 7-10 November 2005 – ASTM E08, "Fatigue and Fracture" & E28, "Mechanical Testing" Committee Meetings, Dallas, TX

SUPERCONDUCTING MATERIALS

Dr. H. Wada
Tsukuba Magnet Laboratory
National Institute for Materials Science
3-13 Sakura
Tsukuba, Ibaraki 305-0003, Japan
Tel: +81 298 59 5024
Fax: +81 298 59 5023
e-mail: WADA.Hitoshi@nims.go.jp

Summary of activities

The TWA 16 annual meeting was held, in conjunction with IEC/TC90, in Niigata, Japan on November 24, 2004 with the participation of 26 people from 5 countries. On-going projects and new proposals were reported and discussed. There are currently eight projects underway.

WG4 started a new project, WG4-2 "Measurement method for tensile property of Nb_3Sn wires at room temperature" to confirm, through a round robin test (RRT), the reliability of the standard test method for tensile property of Nb_3Sn wires at room temperature, which was drafted by the IEC/TC90 WG5 group. The RRT started in March 2005 and will be finished in a couple of months.

The 1906 Award of the IEC was awarded to four of TWA 16's members; H. Wada (Japan), M. Thoener (Germany), T. Matsushita (Japan) and K. Funaki (Japan), for their contributions to the development of IEC standards through VAMAS activities.

Status of current projects

(1) WG1-1: Bending strain effects on critical current in oxide superconductors

Project objectives:
To establish a standard measurement method for the critical current of Bi-2212/-2223 oxide superconductors as a function of bending strain. Critical currents of Bi-2212/-2223 oxide superconductor specimens are measured at 77 K with no external field, after bending each of the specimens to a different, designated curvature at room temperature.

Progress:
Based on the first supplementary test (mini-RRT #1) results that showed smaller critical current degradation against bending strain and less data scatter in the critical current values than that in the VAMAS-RRT, the second supplementary test (mini-RRT #2) was carried out by the same group. In this test, following three methods were tried to reduce the thermal strain in the test sample; (1) lowering soldering temperature by using a low melting point solder, (2) slow cooling (less than 10 K/min) from room temperature to the measuring temperature (77K) and (3) reducing the number of thermal cycles. However, notable differences in critical current degradation and data scatter compared with the VAMAS-RRT result were not observed, indicating that the methods tried were less effective at reducing the thermal strain. Based on results of the VAMAS-RRT and supplementary tests, a draft for bending strain effect measurement will be prepared and proposed to IEC/TC90.

(2) WG1-2: Measurement method for critical temperature in oxide superconductors

Project objectives:
To establish and standardize the measurement method for critical temperature of oxide superconductors.

Progress:
An IEC standard draft for critical temperature measurement method (IEC 61788-10) was proposed as a committee draft at the IEC/TC90 meeting in March 2000. After some modifications on the scope, definition and measurement details, the international standard was published in June 2002.

(3) WG1-3: Measurement method of the irreversibility field in oxide superconductors

Project objectives:
To clarify the relation between V-I characteristics and flux pinning, determine the relation among irreversibility (critical) fields obtained by different measurement methods, and establish an adequate and reliable measurement method. At the moment, the project is concentrating on establishing a standard measurement method for the irreversibility field of bulk YBaCuO superconductors. The critical current density is measured from the hysteresis of magnetization using a SQUID/VSM magnetometer. The irreversibility field is defined by a magnetic field at which the critical current density is reduced to a certain criterion.

Progress since last report:
It is planned to start the round robin testing in the summer of 2005.

(4) Project number; WG1-4 Coupling loss measurement in multifilamentary HTS superconductors

Project objectives:
To examine the adaptability of the VSM method to measure the coupling loss of multifilamentary HTS superconducting tapes.

Progress since last report:
At the TWA 2001 meeting, Dr. Collings proposed a round-robin test (RRT) to measure the coupling loss of LTS and HTS tapes as a function of field ramp rate up to 4 T/min. However, we had no further progress due to difficulty in finding samples and participants. Therefore the project leader proposed at the 2003 TWA meeting a new modified plan, which aimed to establish the measurement method of hysteresis loss of tape samples such as Y-123 coated conductors by pick-up coil and/or VSM methods. The outline of a RRT was presented and discussed at the 2004 TWA meeting. Samples for the RRT are available from a US company.

(5) WG2-1: Measurement methods of trapped field and levitation force in bulk oxide superconductors

Project objectives:
To establish and standardize the measurement methods of trapped flux density and levitation force of HTS bulk superconductors. 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.

Progress since last report: The standard methods for measuring trapped flux density were approved by all the members based on the results of round robin tests. However, the problem as to the extrapolation was raised. The trapped flux density strongly depends on the sample quality, and thus the estimation of the trapped flux density causes serious errors when one assumes that the sample properties are uniform throughout the entire sample. We will continue further round robin tests using new samples, since one of the samples showed some degradation in its field trapping ability during the tests.

An IEC standard draft (IEC 61788-9) for the measurement of trapped flux density was proposed at the IEC/TC90 committee meeting is now at the FDIS stage and circulated among international members for voting.

(6) WG3-1: Measurement methods of surface resistance in thin film superconductors

Project objectives:

To confirm that the accuracy should be 20 % or less for the measurement method for surface resistance of thin film superconductors adopted by the IEC standard, IEC 61788-7. The surface resistance is determined by applying a microwave signal to a cylindrical dielectric resonator sandwiched by two superconducting thin films and measuring the insertion attenuation of the resonator at certain frequencies from 8GHz to 30 GHz.

Progress since last report:

Development of the IEC standard, IEC 61788-7 (published Jan. 2002) has been discussed. The main part of the development of the standard includes: 1) closed type resonators are recommended from the viewpoint of the stable measurements, 2) uniaxial-anisotropic characteristics of sapphire rods are taken into consideration for designing the size of the sapphire rods, and 3) recommended measurement frequencies of 18 GHz and 22 GHz are added to the current 12 GHz measurement. The possibility of a new international RRT in which the same superconductor films are measured by each participating institute at different frequencies will be considered.

(7) WG4-1: Measurement method for the mechanical properties of oxide superconductors

Project objectives:

To establish and standardize the measurement method for mechanical properties such as yield strength and Young's modulus of oxide superconductors as well as MgB_2 composite tapes. The measurement is performed at room temperature as well as at cryogenic temperatures using a mechanical test machine.

Progress since last report:

WG4 is trying to elucidate causes of the large scatter in Young's modulus and elongation obtained among participants at the 1^{st} RRT of a room temperature tensile test for Bi2223 tape samples. It was found important to take into consideration the initial strain state of each of components, i.e., silver sheath, silver alloy sheath and Bi2223, for the analysis of a stress-strain curve. At the 2004 TWA meeting, it was decided to provide a new draft for testing mechanical property of Ag/Bi2223 tapes. By using this new draft, we plan to implement a RRT in near future.

The revised draft of IEC61788-6 ed. 2 (room temperature tensile test of composite superconductors) covering Cu/NbTi wires, Cu/Nb_3Sn wires and Ag/Bi2223 tapes was prepared for submission to IEC-TC90 meeting in October, 2004. At that meeting, it was decided to separate the standard to three different standards each covering Cu/NbTi wires, Cu/Nb_3Sn wires and Ag/Bi2223 tapes. At present a group composed of German and Japanese experts are writing a new draft only for Ag/Bi2223 tapes.

(8) WG4-2: Measurement method for tensile property of Nb_3Sn wires at room temperature

Project objectives:
To confirm, through a RRT, the reliability of the standard test method for tensile property of Nb_3Sn wires at room temperature, which was drafted by the IEC/TC90 WG5 group.

Progress since last report:
The draft (Nb_3Sn-WG5- 50316) of the test method was proposed by the German members of IEC/TC90 WG5 in November 2004, partially modified in the WG5 group and completed in March 2005. The RRT, initiated March 2005, was planned to confirm the reliability of the drafted test method. Common samples of Nb_3Sn wires for the RRT were supplied by the four companies; EAS, Hitachi Cable, Kobe Steel and Mitsubishi Electric. The RRT is expected to finish in May 2005. Soon after the completion of the RRT, the results will be transferred to the IEC/TC90 WG5 group for standardization.

Technical Working Area 17

CRYOGENIC STRUCTURAL MATERIALS

Dr. T. Ogata
National Institute for Materials Science
1-2-1, Sengen
Tsukuba-shi, Ibaraki 305-0047, Japan
Tel: +81 298 59 2541
Fax: +81 298 59 2501
e-mail: OGATA.Toshio@nims.go.jp

General overview of activities

Results of three projects, "Interlaminar Shear test on GFRP", "Mechanical tests in high magnetic field" and "Advanced fracture toughness test" were discussed and drafts of TTA documents on those projects have been revised.

The standard in ISO/TC164/SC1 "Metallic Materials - Tensile Testing in Liquid Helium", ISO 19819 was published in August 2004.

A round robin test on Young's modulus measurement during tensile test in liquid helium was carried out.

A new project, Tensile Test for Composite Materials at Cryogenic Temperatures, was initiated.

Status of Current Projects

Project No. 5, Interlaminar Shear test on GFRP

Project objectives:
Develop an understanding of mechanical property determination by interlaminar shear tests of GFRP at liquid helium temperature and establish a unified and reliable testing method through a series of RRT.

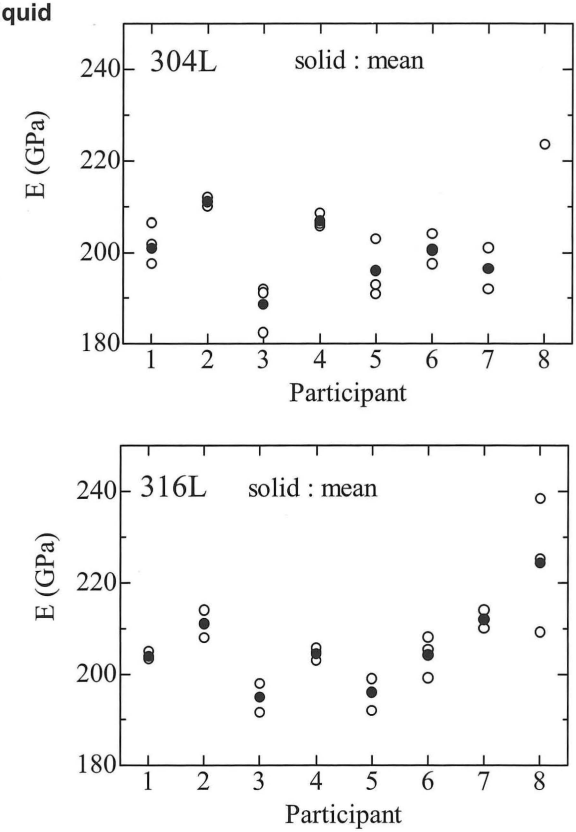
Progress since last report:
A TTA document for ISO/TC 61 is being revised, after receiving comments from TWA 5 members.

Project No. 8, Young's modulus measurement during tensile testing in liquid helium:

Project objectives:
Develop an understanding and testing method for Young's modulus measurement during tensile test in liquid helium and establish a testing method through a series of RRT.

Progress since last report:
In February, LINDE reported new data obtained from additional tests. After considering this new data, the standard deviation of Young's moduli increased from 4.6 GPa to 5.5 GPa (from 2.3% to 2.7% of the mean values of 202 GPa and 200 GPa, respectively) for 304L steel and from 4.2G Pa to 4.7 GPa (from 2.0% to 2.3% of the mean values 205 GPa and 204 GPa) for 316L steel.

Figure 1. Results of round robin test on Young's modulus measurement during tensile test in liquid



Project No. 9, Tensile Test for Composite Materials at Cryogenic Temperatures

Project objectives:
Develop an understanding of in-plane tensile property determination of GFRP at cryogenic temperatures and establish a unified and reliable testing method through a series of round-robin tests (RRT).

Progress since last report:
Three types of specimens were prepared, and a series of tensile tests was conducted at room temperature, liquid nitrogen temperature (77 K) and liquid helium temperature (4 K) to investigate the effects of temperature and specimen geometry on the in-plane tensile properties of glass-cloth/epoxy laminates. The RRT programs to evaluate testing procedures for cryogenic tensile properties have been planned.

Other standardization activities

A proposal of the standard in ISO/TC164/SC1 "Metallic Materials - Tensile Testing in Liquid Helium" which was submitted on January 2000 has been published in August 2004 as ISO 19819.

Technical Working Area 18

STATISTICAL TECHNIQUES FOR INTERLABORATORY STUDIES

Dr. J. Polzehl
Weirstrass Institute for Applied Analysis and Stochastics
Mohrenstr. 39, D-10117 Berlin, Germany
Tel: +49 30 20372 481
Fax: +49 30 2044975
e-mail: polzehl@wias-berlin.de

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. In general it is not intended that the TWA initiates its own projects. During the past year TWA18 has reviewed four project proposals from TWAs 20, 22, 25 and 29. 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

Prof. G. Webster
Imperial College of Science, Technology and Medicine
London, SW7 2BX, UK
Tel: +44 20 7594 7080
Fax: +44 20 7594 7017
e-mail: g.webster@ic.ac.uk

Project 1: Measurement of Residual Stress by Neutron Diffraction

The first project is concerned with the measurement of residual stress by neutron diffraction and is now reaching its conclusion. Recent activity has been concerned mainly with editing the draft technical specification that has been prepared by the joint ISO/TC135 and CEN/TC138 AHG7 committee. The edited draft has been distributed to National bodies and has been approved by 24 members with no objections. It is to be issued as prCEN ISO TS 21432 *Non-destructive testing – Standard test method for determining residual stresses by neutron diffraction.*

For a case study of this TWA see the Feature Article *VAMAS TWA 20 Measurement of Residual Stress Case Study* in this VAMAS Bulletin.

Progress on the measurement of residual stress at the new engineering diffractometers SMARTS in Los Alamos and ENGIN-X at ISIS have been reported at the 2nd American Conference on Neutron scattering (ACNS) in College Park, Maryland in June 2004. A facility, FaME 38 at ILL, is now fully operational for providing advice on making residual stress measurements by neutron and synchrotron diffraction methods. A symposium was held on 27 April 2005 at TWI, Cambridge to discuss the role of residual stress on structural integrity.

Project 2: Measurement of Residual Stress by High Energy Synchrotron X-ray Diffraction

The aims of this project, started in September 2003, are to:

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

Two round robin samples (an aluminium alloy weld in the form of a thick piece and thin slice and a titanium alloy laser shock peened plate) have been prepared. Protocols for making measurements have been drafted and measurements started in both round robin specimens. The next full project meeting is to be held after MECASENS III in Santa Fe on 20 October 2005.

Project 03: Measurement of Residual Stress by X-ray Diffraction

The main objectives of this project, approved in 2004, are to coordinate the XRD activity, examine various aspects of the measurement process, and validate and contribute to an internationally agreed standard that is currently being developed through CEN/TC138/WG10 and within the ASTM E28.13 subcommittee on Residual Stress Measurement.

Stainless steel specimens in the form of a ring and plug with well-characterised residual stress distributions are being prepared for an intercomparison, together with beam samples for elastic constant determinations.

MECHANICAL MEASUREMENTS FOR HARDMETALS

Dr. B. Roebuck
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex, TW11 0LW, UK
Tel: +44 20 8943 6298
Fax: +44 20 8943 2989
e-mail: bryan.roebuck@npl.co.uk

Current project: Toughness Tests for Hardmetals

The toughness project was aimed at developing good practice in the toughness testing of hardmetals. Nine laboratories from 7 countries participated in testing, with correspondence support from a further 5 laboratories in 3 more countries. 10 batches of material from 4 organisations were supplied for the evaluation of four test methods; the chevron notch short rod, the Palmqvist test (where crack lengths at indentation corners are measured), and two versions of a notched beam test. A full set of samples were circulated to participants in autumn 2001. A first preliminary report was written and circulated to participants in mid 2004, a technical presentation was discussed at a one-day seminar at NPL in February 2004, and a paper was given at an international Powder Metallurgy Conference in Vienna in October 2004. The report was also tabled at the ISO committee meeting in Vienna in October 2004. Following feedback and further revision a final report was issued in March 2005.

Regular progress reports were given to

- ISO TC119/SC4 on Cemented Carbides/Hardmetals (most recent meeting in Vienna, Austria in October 2004).
- EHMG, European Hard Materials Group (part of EPMA, European Powder Metallurgy Association) – usually meets every 6 months or so.
- BHECTA, Research Group of British Hardmetal and Engineers Cutting Tool Association – meets every 3 months. Members also include VAMAS participants from USA and Germany.
- Recipients of NPL Hard Materials Newsletter – database of > 400 worldwide.

TWA discussion groups are usually held in association with International Powder Metallurgy conferences and meetings. The 2005 calendar includes:

- EPMA Hard Materials Group Meeting, Plansee Seminar, Reutte, Austria, May 2005.
- European PM conference, Prague, October 2005.
- ISO TC119/SC4 Meeting in Prague in October 2005.

Future Projects

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 was discussed at a recent ISO meeting in Vienna, Austria in October 2004. Preliminary work was conducted by a UK/European subgroup of the VAMAS contact group and issued as an NPL Measurement Note. This has been submitted for discussion at the next ISO meeting in October 2005 (probably Prague).

Another possible project area could include technical support for a standard on grain size measurements. NPL has prepared a new draft standard and the VAMAS group will be asked to review the ISO CD (Committee Draft). This would be a paper exercise, with no underpinning technical work involved.

Technical Working Area 22

MECHANICAL PROPERTY MEASUREMENTS OF THIN FILMS AND COATINGS

Dr. N Jennett
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex, TW11 0LW, UK
Tel: +44 20 8943 6641
Fax: +44 20 8614 0451
e-mail: nigel.jennett@npl.co.uk

VAMAS Technical Working Area 22 addresses pre-standardization needs in the general area of test method development and evaluation for the measurement of the mechanical properties of thin films and coatings on substrates. Activities cover measurement methods to determine the mechanical properties of the films or coatings themselves, as well as the performance of coating/substrate systems as a whole. There are currently three approved projects underway in TWA 22, two projects having been completed in the past year.

Project 1: Measurement of Hardness and Young's Modulus of Thin Coatings Using Depth Sensing Indentation Instruments

Project 1, a large round-robin exercise to evaluate the accuracy of instrumented indentation testing (IIT) to determine hardness and elastic modulus of thin coatings, has been completed. All data have been collected, analyzed and compiled, and a detailed report has been prepared by the project leaders at NPL.

Project 2: Adhesion of Thin Coatings

Intercomparison testing for Project 2, on adhesion of CrN coatings on AISI 304 SS, with and without gold-palladium interlayers, has been completed, and the results written up for publication.

Project 3: Elastic Properties of Thin Films and Coatings

In this project, a comparison of two complementary techniques for the determination of elastic properties of thin films is underway, led by Uwe Beck at BAM. Several mini-round-robins have been conducted to confirm the suitability of specific film-coating systems for testing by IIT and surface acoustic wave spectroscopy (SAWS).

Project 4: Measurement of Super-hard Coatings by Instrumented Nano-indentation

Very hard materials, particularly as thin films on substrates, are finding many new product applications, but the accurate measurement of their hardness poses a unique set of challenges for IIT. This project will test ISO/CD 14577, Part 4 'Test method for coatings' and determine its applicability to the measurement of super hard materials that possess a very high elastic moduli and high yield stress. The project will also establish the relationship between the microstructure of a super hard material and its eventual properties, in particular modulus and hardness.

Project 5: Instrumented Indentation Testing intercomparison: (ISO14577 – part 4)

This project was approved earlier this year. Its objectives are to:

- Collaborate with a NIST intercomparison to test competence to measure copper on Si by nano-indentation.
- Compare performance of TWA22 participants in using ISO/DIS 14577 part 4 'Test method for coatings' to perform the NIST requested measurements.
- Compare the results obtained by ISO14577- part 4 procedures with those obtained by the single depth indentation procedure suggested in the NIST intercomparison.
- Input to development of the standard ISO 14577 Part 4 Test method for coatings.

PERFORMANCE RELATED PROPERTIES FOR ELECTROCERAMICS

Dr. M. Cain
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex, TW11 0LW, UK
Tel: +44 20 8943 6599
Fax: +44 20 8943 2989
e-mail: markys.cain@npl.co.uk

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

This project is complete with a VAMAS report having been published: VAMAS Report 47 (2004).

Project 2: ***Measuring d_{33} from displacement dependence of electric field strength.***

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 carry out an International Interlaboratory exercise on the measurement of polycrystalline materials and the new single crystal piezoelectric materials. All samples have now been sent out by the Japanese leaders, and results are starting to be returned.

Project 3 Title: ***International intercomparison of the measurement of the polarization loop response for ferroelectric thin films.***

Ferroelectric thin films form an integral part of FRAMs (Memories) and many solid-state sensors and some actuators. Their properties are affected by geometry, composition, structure, driving voltages etc. In this VAMAS project, one of the most basic of tests will be explored in an effort to elucidate measurement good practice. The round robin will determine the experimental variability and limitations in the measurement of the ferroelectric polarization – electric field (PE) loop response for ferroelectric thin films by the measurement of charge and voltage. The first phase of the project was completed in 2004. Responses from experts in this field have highlighted the need to run a second Interlaboratory comparison adopting identified measurement best practice.

Dissemination activities

Dissemination and awareness of TWA 24 continues through external collaborations and conference presentations. Meetings and dissemination activities were spread across Europe, Japan and America. Specifically in 2004-2005 the following activities included reference to this TWA:

- EC Thematic Network: POLECER, Polar Electroceramics, with separate tasks focused on standardization issues. The web pages are continuingly being expanded to include more information about VAMAS.
- Electroceramics 9, France, June 2004. Research on the Berlincourt method was described to an international audience with direct relevance to VAMAS project No.1.
- IFFF 2004: International Conference in Korea, April 2004. Several presentations were made relating to TWA 24 activity.
- EC POLECER Network - Symposium: Winter Workshop – integrated devices, Chateaux D'Or, Switzerland; presentation on PFM method with reference to VAMAS.
- EC MIND – New Network of Excellence, starting March 2005. M G Cain (NPL) introduced the concepts behind the development of measurement best practice by close alignment with VAMAS, thus testing new ideas prior to new standards in this emerging scientific and technological discipline. This strategy was welcomed by the MIND committee and EU commissioner at a meeting in Copenhagen on 3-4 March 2005.

Proposed future projects and TWA directions

The continuing need for pre-normative standards activity in the field of electroceramics is exemplified by various market drivers and legislative issues surrounding their application or use. Examples include the use of piezoelectrics in automotive fuel injectors, used at very high pressures, temperatures and electrical fields, all of which are currently not described in any international standards documents. Another example may be in the use of electroceramic thin or thick films that currently suffer from a lack of standards. Applications here include MEMS devices, sensors and memory devices. The measurement of loss and issues surrounding energy efficiency are also not dealt with in existing standards and much work is needed to understand these issues before new standards may be written. A new social driver is that to reduced the amount of lead in the environment. So far, most piezoelectric materials are based on lead-containing ceramic compounds – taken by most to be inherently safe. However, with the development and patenting of new lead-free compositions (notably by Toyota and Denso, Japan), then this TWA will start to explore these issues on an international basis. TWA 24 is perfectly aligned with industrial needs in these and other areas, some of which are the subject of the proposed projects below which are at an early planning/discussion phase.

- Residual stresses in thin films
- Finite element analysis and data provision
- Thin film measurement methods – via EU NoE MIND
- Single crystal nomenclature – via POLECER
- Measurement of piezoelectric and dielectric properties at high stress
- Measurement of electrical and mechanical fatigue of piezoelectric ceramics materials
- Properties of electrical conductive, optical transparent thin films
- Thermal effects on performance
- The dielectric, elastic and piezoelectric properties (matrix elements) need to be measured as complex coefficients 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.
- The temperature dependence of the complex coefficients is important in some applications e.g. high power and space applications.
- Hydrostatic property measurements to about 14 MPa are important for underwater applications.
- Clarification of the meaning of fatigue / aging / degradation, particularly the distinction between reversible and irreversible mechanical or electrical damage
- In project 1, to introduce measurements of piezoelectric coefficient of thin and thick films on substrates.

The TWA 24 home page is sited within the Functional Materials Group Website at NPL: http://www.npl.co.uk/npl/cmmt/functional/vamas.html

Technical Working Area 25

CREEP/FATIGUE CRACK GROWTH IN COMPONENTS

Dr. K. Nikbin
Imperial College
Mechanical Engineering Department
London, SW7 2BX, UK
Tel: +44 20 7594 7133
Fax: +44 20 7823 8845
e-mail: k.nikbin@ic.ac.uk

Overall Aims of TWA 25

- Use of the recently published data from BRITE/EURAM European collaborative programmes (CCG in C-Mn, HIDA, LICON, CRETE) in VAMAS TWA 25 round robin review of industrial test methods in order to establish the relevant specimen geometries and the testing and analysis methodology.
- Establish and validate results from a crack growth prediction round robin of pipe and a plate tests carried out at elevated temperatures.
- Validate results against measurements on standard laboratory specimens using the ASTM E1457-2001 testing procedure and the new European Code of Practice for creep crack growth testing of laboratory geometries.
- Produce a document containing a list of generic component geometries and the associated parameters which can be used in creep/fatigue crack growth testing of components.
- Recommendations in a draft Code of Practice for dealing with component creep/fatigue crack growth testing and analysis of laboratory and industrial feature specimens

Present and Future Activities

During this period VAMAS TWA 25 has accomplished its planned objectives. Table 1 gives the list of the completed tasks and the outputs provided by the work. There are a number of disseminated documents and published papers. This information will be used to develop the next stage of the project and to finally produce a draft Code of Practice for Creep/Fatigue Component testing.

The substantial industrial interest in this project and participants in different countries has translated into 18 paper contributions towards a special technical publication in the International Journal of Pressure Vessel Piping (Vol. 80, Issues 7-8, Pages 415-595 (July - August 2003) *Creep crack growth in components* Edited by: K. M. Nikbin).

As a follow up to the present TWA 25 it has been planned to continue the work on developing a Code of Practice for Welded Components. This is in collaboration with a number of European and International committees such as ASTM, TC11, NET. There is wide interest in industry to be involved in this project as weld related problems is seen to be of vital importance.

Table 1: TWA 25 Crack/Fatigue Crack Growth in Components

Work tasks	Completion date	Output
Collect shared information	Nov 1999 to Nov 2000 COMPLETED	Data-base - collected
Round Robin review of industrial test methods	Jan. 2002 COMPLETED	Review Document – completed and disseminated
Round Robin crack growth prediction of a pipe and a plate	Dec. 2001 to Dec. 2002 COMPLETED	Document – of different analysis methods
Establish appropriate list of specimen geometries and testing and analysis methodology	June 2002 to May. 2003 COMPLETED	Document – annex in a draft Code of Practice
Publication of a special issue in the Int. J. of Pressure Vessel and Piping	June-2002 to Sept. 2003 COMPLETED	Journal publication in IJPVP of the partners findings: 18 papers
Draft Code of Practice for Creep/fatigue testing of components	May 2004 COMPLETED	Draft Code of Practice. ISO document for Notch bar tests complete and distributed. British Energy TFAD procedure distributed
Round Robin Testing of Weld and HAZ specimens	Jan 2005 to Dec. 2007	Some specimens distributed Application for a New TWA number
ASTM Papers	May 2005	

Other Planned Dissemination Activities in the Coming Year:

- ASME – Residual stress meeting July 2005
- Discussion with ISO to develop the Code of Practice into a ISO/TTA document.
- Collaborate with API, ASME and ASTM E08 Creep fracture committee to share information
- Collaboration with ASTM. TC11, FITNET, WELDON, NET European network on neutron techniques for residual stresses

FULL FIELD OPTICAL STRESS AND STRAIN MEASUREMENT

Dr. R. Burguete
Airbus UK Ltd
Filton, Bristol, BS99 7AR, UK
Tel: +44 117 936 4299
Fax: +44 117 936 4344
e-mail: richard.burguete@airbus.com

and

Prof E. A. Patterson
Michigan State University
Department of Mechanical Engineering
2555 Engineering Building
East Lansing, MI 48824-1226, USA
Tel: +1 517 355 5131
Fax: +1 517 353 1750
e-mail: eann@egr.msu.edu

Summary of Progress

The EU project Standardisation Project for Optical Techniques of Strain Measurement (SPOTS) is well underway, and is the basis for the VAMAS TWA 26 project.

The detailed scientific and technical objectives are:

1. The development of physical reference materials, reference geometry, and reference loading conditions for image correlation, ESPI, shearography, moiré, photoelasticity, and thermoelasticity.
2. The design and construction of simulated, virtual reference materials as a first step. For each technique, the virtual reference materials will include: simulated data, synthesised fringe patterns with and without random or systematic noise.
3. Definition of recommended data formats for image data, numerical data, and processed data for full-field optical techniques of strain measurement.
4. Optimisation of methodologies for the use of unified reference materials and for the practical application of image correlation, ESPI, shearography, moiré, photoelasticity, and thermoelasticity.
5. Liaison with international bodies associated with optical methods of strain measurements, in order to ensure recognition of the measurement procedures and reference materials developed.

6. Identification of routes for traceability for calibration of systems.

The major technical achievements of the project will be a unified approach to the calibration and comparison of optical systems for measuring full-field strain distributions. Since this is not a readily quantified output, a work package has been designed to generate data that will demonstrate the efficacy of these achievements. The tangible achievements will be the definition of recommended data formats; development of physical and virtual reference materials; optimised methodologies for the use of the reference materials, and for the application of the optical techniques. These outputs will be promulgated via the project website and will be actively disseminated to the EU industrial base through publication in the trade and professional literature, a presence at major conferences and exhibitions and through liaison with industrial and professional bodies. These techniques have been identified as those of most interest to industry.

CHARACTERIZATION METHODS FOR CERAMIC POWDERS AND GREEN BODIES

Dr. R. Wäsche
Federal Institute for Materials, Research and Testing (BAM)
12205, Berlin, Germany
Tel: +49 30 8104 1541
Fax: +49 30 8104 1547
e-mail: rolf.waesche@bam.de

Determination of coarse particles in advanced ceramic powders

The first project, "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. An international round robin was carried out. The participation included 10 different partners from 6 countries

- Germany
- Italy
- Japan
- Korea
- Thailand
- USA

The results of the round robin were discussed during the TWA 27 meeting in Kurashiki, Japan, in September 2003.

The project is related to ISO TC 206 PWI 03 "Determination of content of coarse particles in ceramic powders by wet sieving method". During the ISO TC 206 meeting, held at Nagoya on October 2.-3, 2003, the working draft on determination of coarse particles was accepted and registered as ISO/DIS 24369. The corresponding ISO working group accepted the proposed suggestions made by VAMAS TWA 27. The improvements were based on the results of the VAMAS round robin concerning the corrections made to the measuring procedure.

The project on determination of coarse particles has now been completed.

Future Project

Reliability and accuracy of particle size measurements in medium to high concentrated suspensions using acoustic spectroscopy

Determination of particle size of ceramic powders in the nanometer range is an important task nowadays in both production technology and in materials metrology. Such measurements are not only limited by available methods but also by a lack of results confirming their accuracy and reliability. Optical methods, i.e. Laser Diffraction and conventional Photon Correlation Spectroscopy (PCS), are both unsuitable for accessing nanometer range particle distributions for the following reasons. Laser Diffraction is limited to the micron size range by its physical principle. Conventional PCS can be used only in very diluted suspensions. These limitations can be overcome by Ultra Sound Spectroscopy which is not limited by size of the particles nor by the concentration of the suspension up to about 30 vol%.

The objective of the proposed project is to verify the accuracy and repeatability of particle size measurements in the lower sub-micron and nano size range by comparative interlaboratory testing.

The **next TWA meeting** is due to be held on Sept. 15, 2005, during the PacRim 6 meeting, Maui, Hawaii.

Technical Working Area 28

QUANTITATIVE MASS SPECTROMETRY OF SYNTHETIC POLYMERS

Dr. C. Guttman
National Institute of Standards and Technology
Gaithersburg, MD, USA
Tel: +1 301 975 6729
Fax: +1 301 975 3928
e-mail: charles.guttman@nist.gov

TWA 28 currently has one project, 'Method Development for Matrix Assisted Laser Desorption/Ionization (MALDI) Time of Flight (TOF) Mass Spectrometry (MS) of Low Molecular Mass Polystyrene (PS)'.

Mass spectrometry is a standard method for mass analysis of low mass organic molecules. However, only recently has a type of mass spectrometry, MALDI MS, been applied to synthetic polymers. The use of a different type of mass spectrometry and its application to high mass polymers possessing a distribution of masses imply that the current standard methods of analysis do not apply. The current project was undertaken to address the need for a standard method for analysis of synthetic polymers by mass spectrometry. A method for the use of MALDI MS for the molecular mass distribution (MMD) determination of polystyrene (PS) of molecular masses from 2000 u to 35000 u has been written following that of the DIN or ASTM method for the determination of the MMD of PS by an alternate technique, gel permeation chromatography, (GPC). Measurement specifics are based on the protocol used in the recent interlaboratory comparison of PS by MALDI MS, organized under the auspices of the American Society of Mass Spectrometry (ASMS).

Current members of the project include:

- NIST Polymers Division, USA
- University of Alabama-Birmingham, USA
- PPG Industries, USA
- AIST, Japan
- University of Alberta, Canada
- ICI, England
- University of Tennessee, USA
- University of Catania, Italy
- Union Carbide Corp., USA
- BAM, Germany

The VAMAS TWA 28 meeting was held at the ASMS 52nd Annual Meeting on Mass Spectrometry and Allied Topics in Nashville, June 2004, following the Polymeric Materials Interest Group meeting. Standardisation of the procedure is being considered in ISO, DIN and ASTM. In November 2004 the draft method received subcommittee approval from ASTM D20.70 the subcommittee on Physical Methods in Polymer Science. In January of 2005 the method was submitted to ballot to D20 on Plastics to be accepted as an ASTM method. The method now awaits final ASTM D20 approval.

Members of the TWA who attended the June 2005 meeting of the American Society of Mass Spectrometry in San Antonio TX, USA discussed the revised method, the review of the test methods from the standards developing organizations, and work on round robin plans to extend the Method as requested by ASTM.

Future Meetings Relevant to VAMAS Project:

A meeting will also be held at the annual NIST Polymer MS Workshop in November of 2005 in Gaithersburg, MD.

Technical Working Area 29

MATERIALS PROPERTIES ON THE NANOSCALE

Dr David Mendels
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex
TW11 0LW, United Kingdom
Tel: +44 20 8943 6206
e-mail: david.mendels@npl.co.uk

Dr Stephen Hsu
Room A-263, Building 223
National Institute of Standards and Technology
Gaithersburg, MD 20899
USA
Tel. +1 301 975 6120
Fax +1 301 990 8729
e-mail: stephen.hsu@nist.gov

The TWA was approved at the Steering Committee meeting in May 2003, Petten, The Netherlands. The objectives of the TWA were stated as:

- To review the current state of the art in measuring and characterization of material properties at the nanoscale. In a first step, the main interest will be on nanomechanical properties.
- To establish a classification of nanomaterials and material systems at the nanoscale.
- To compile and compare international measurement methods that currently exist.
- To develop new measurement methods for characterizing physical and mechanical properties of materials with at least one length scale of dimension less than 100 nm. This includes both hard and soft materials.
- To provide recommendations for good practice on materials characterization at the nanoscale.

Preliminary work on the first project has commenced, and a proposal has been submitted to the SC for approval.

Status of Current Project:

Protocols for the Calibration of the Force Constant for Scanning Probe Methods

The scientific goal of this project is to develop appropriate methods for the measurement of the force constant of indentation-based methods. This work underpins most materials nano-metrology areas, in particular MEMS, MOEMS, NEMS and quantitative force measurement phenomena at the molecular level.

In this study:

- AFM cantilevers and V-shaped probes will be tested in a round-robin for Force calibration in the pN through to µN range;
- An initial pre-study, restricted to vertical spring constant calibration, will be carried-out between main participants to determine the appropriate procedures of handling and exchanging probes;
- A full round-robin study will be carried-out on both vertical and torsional stiffness without restrictions on the method used to calibrate the probe.
- Based on the results, a best practice guide including recommendation for the test operation and a description of the methods used will be produced, and used as a starting point for further activity on the most successful methods.

Initial intercomparison work involving NPL, NIMS, NIST and BAM has highlighted difficulties in handling AFM cantilevers and V-shaped probes, with high damage rates to specimens occurring while they are being shipped and cleaned before measurement. This has implications for executing a larger intercomparison study, as it is now clear that specials procedures need to be adopted.

Proposed/Future Projects and TWA Directions

- Tip shape characterisation for AFMs, SPMs and nano-indenters
- Mechanical properties of soft materials at the nano-scale
- Nanoindentation of compliant multi-layered system

• VAMAS Technical Working Areas •

Technical Working Area 1
Wear Test Methods
Dr. Erich Santner
BAM
Unter den Eichen 44-46
D-12200 Berlin, Germany
Tel: +49 30 8104 1810
Fax: +49 30 8104 1817
erich.santner@bam.de

Technical Working Area 2
Surface Chemical Analysis
Dr. Cedric Powell
Mail Stop 8370
NIST
Gaithersburg, MD USA 20899
Tel: +1 301 975 2534
Fax: +1 301 216 1134
cedric.powell@nist.gov

Technical Working Area 3
Ceramics for Structural Applications
Dr. Kristin Breder
Saint-Gobain Abrasives
Higgins Grinding Technology Center
1 New Bond St.
Box 15008, MS 413-201
Worcester, MA, USA 01605-0008
Tel: +1 508 795 4147
Fax: +1 508 795 4283
Kristin.Breder@Saint-Gobain.com

Technical Working Area 5
Polymer Composites
Dr. Graham D. Sims
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex
TW11 0LW, United Kingdom
Tel: +44 20 8943 6564
Fax: +44 20 8614 0433
graham.sims@npl.co.uk

Technical Working Area 10
Computerized Material Data
Dr. Kohmei Halada
NIMS, Tsukuba Laboratories
1-2-1 Sengen, Tsukuba-shi
Ibaraki 3050047, Japan
Tel: +81 298 59 2352
Fax: +81 298 59 2301
HALADA.Kohmei@nims.go.jp

Technical Working Area 13
Low Cycle Fatigue
Dr F A Kandil
EuroTest Solutions Limited
PO Box 1226, Kingston,
UK, KT2 5YJ
Tel: +44 20 8541 5244
Fax: +44 20 8541 5520
fathy.kandil@eurotest-solutions.co.uk

Technical Working Area 16
Superconducting Materials
Dr. Hitoshi Wada
Graduate School of Frontier Sciences,
University of Tokyo, 5-1-5 Kashiwanoha,
Kashiwa, Chiba 277-8561, Japan
Tel & fax: +81-4-7136-3781
wada.hitoshi@k.u-tokyo.ac.jp

Technical Working Area 17
Cryogenic Structural Materials
Dr. Toshio Ogata
NIMS, Tsukuba Laboratories
1-2-1, Sengen, Tsukuba-shi
Ibaraki 3050047, Japan
Tel: +81 298 59 2541
Fax: +81 298 59 2501
OGATA.Toshio@nims.go.jp

Technical Working Area 18
Statistical Techniques for Interlaboratory Studies
Dr. Jörg Polzehl
W I A A S
Mohrenstr. 39
10117 Berlin, Germany
Tel: +49 30 20372 481
Fax: +49 30 2044975
polzehl@wias-berlin.de

Technical Working Area 20
Measurement of Residual Stress
Prof. George A. Webster
Imperial College of Science, Technology & Medicine
Exhibition Road, London
SW7 2BX, United Kingdom
Tel: +44 20 7594 7080
Fax: +44 20 7594 7017
g.webster@ic.ac.uk

Technical Working Area 21
Mechanical Measurements for Hardmetals
Dr. Bryan Roebuck
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex
TW11 0LW, United Kingdom
Tel: +44 20 8943 6298
Fax: +44 20 8943 2989
bryan.roebuck@npl.co.uk

Technical Working Area 22
Mechanical Property Measurements of Thin Films and Coatings
Dr. N Jennett
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex, TW11 0LW, UK
Tel: +44 20 8943 6641
Fax: +44 20 8614 0451
nigel.jennett@npl.co.uk

Technical Working Area 24
Performance Related Properties for Electroceramics
Dr. Markys Cain
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex
TW11 0LW, United Kingdom
Tel: +44 20 8943 6599
Fax: +44 20 8943 2989
markys.cain@npl.co.uk

Technical Working Area 25
Creep/Fatigue Crack Growth in Components
Dr. Kamran Nikbin
Imperial College of Science, Technology & Medicine
Exhibition Road, London
SW7 2BX, United Kingdom
Tel: +44 20 7594 7133
Fax: +44 20 7823 8845
k.nikbin@ic.ac.uk

Technical Working Area 26
Full Field Optical Stress and Strain Measurement
Dr. Richard Burguete
Airbus UK Ltd.
New Filton House
Filton
Bristol, BS99 7AR
United Kingdom
Tel: +44 (0)117 936 4299
Fax: +44 (0)117 936 4344
richard.burguete@airbus.com

Technical Working Area 27
Characterization Methods for Ceramic Powders and Green Bodies
Dr. Rolf Wäsche
BAM
Unter den Eichen 87
D 12200 Berlin
Germany
Tel: +49 30 8104 1541
Fax: +49 30 8104 1547
rolf.waesche@bam.de

Technical Working Area 28
Quantitative Mass Spectrometry of Synthetic Polymers
Dr. Charles Guttman
Mail Stop 8541
NIST
Gaithersburg, MD, USA 20899
Tel: +1 301 975 6729
Fax: +1 301 975 3928
charles.guttman@nist.gov

Technical Working Area 29
Materials Properties on the Nanoscale
Co-chairmen:
Dr David Mendels
Engineering and Process Control Division
National Physical Laboratory
Teddington, Middlesex
TW11 0LW, United Kingdom
Tel: +44 20 8943 6206
david.mendels@npl.co.uk

Dr Stephen Hsu
Room A-263, Building 223
NIST
Gaithersburg, MD 20899
USA
Tel. +1 301 975 6120
Fax +1 301 990 8729
stephen.hsu@nist.gov

• VAMAS Steering Committee •

UNITED STATES

VAMAS CHAIRMAN
Dr Stephen Hsu
Room A-263, Building 223
National Institute of Standards and Technology
100 Bureau Drive,
Gaithersburg, MD 20899
USA
Tel. +1 301 975 6120
Fax +1 301 990 8729
stephen.hsu@nist.gov

VAMAS SECRETARY
Dr Stephanie Hooker
National Institute of Standards and Technology
Materials Reliability Division
325 Broadway
Boulder, CO 80305
00 1 303-497- 4326
FAX: 00 1 303-497- 5030
shooker@boulder.nist.gov

Dr. Stephen Freiman
Deputy Director
Materials Science and Engineering Laboratory
National Institute of Standards and Technology
100 Bureau Drive, Mail Stop 8520
Gaithersburg, MD 20899
USA
Tel: +1 301 975 5658
Fax: +1 301 975 5012
stephen.freiman@nist.gov

Dr. Leslie E. Smith
Director
Materials Science and Engineering Laboratory
National Institute of Standards and Technology
100 Bureau Drive, Mail Stop 8500
Gaithersburg, MD 20899
USA
Tel: +1 301 975 5658
Fax: +1 301 975 5012
mailto:leslie.smith@nist.gov

CANADA

Dr. Blaise Champagne
Director General
Industrial Materials Institute
National Research Council Canada
75 Boulevard de Montagne
Boucherville, Québec J4B 6Y4 , Canada
Tel: +1 514 641 5050
Fax: +1 514 641 5101
blaise.champagne@nrc.ca

Dr. Hamid Mostaghaci
Science and Technology Division (TBR)
Department of Foreign Affairs & Intl Trade
125 Sussex Drive, Room C3-244
Ottawa, Ontario, K1A 0G2, Canada
Tel: +1 613 995 7920
Fax: +1 613 944 2452
hamid.mostaghaci@dfait-maeci.gc.ca

EC

Prof. Hendrik Emons,
Head, Reference Materials Unit
Institute for Reference Materials and Measurements
Joint Research Centre of the European Commission
Retieseweg 111
B-2440 Geel, Belgium
Tel: +32-14-571722
Fax: +32-14-590406
hendirk.emons@cec.eu.int

Dr. Ir. Gert Roebben
Reference Materials Unit
Institute for Reference Materials and Measurements
Joint Research Centre of the European Commission
Retieseweg 111
B-2440 Geel, Belgium
Tel: +32-14-571816
Fax: +32-14-571548
gert.roebben@cec.eu.int

FRANCE

Prof. Claude Bathias
Conservatoire National des Arts et Métiers
Dept de Génie Mécanique
2 rue Conté, Paris 75003
France
Tel: +33 1 40 27 23 22
Fax: +33 1 40 27 23 22 or +33 1 40 27 23 41
bathias@cnam.fr

Prof. Alain Vautrin
Departement Mecanique et Materiaux Centre SMS
Ecole des Mines de Saint-Etienne
158, cours Fauriel,
F-42023 Saint-Etienne Cedex 2
France
Tel: +33 0 47 74 20 190
Fax: +33 0 47 74 20 000
vautrin@emse.fr

GERMANY
Dir. and Prof. Dr.-Ing.
Wolfgang Paatsch
Department VIII.
Bundesanstalt für
Materialforschung und –
prüfung
Unter den Eichen 44-46,
12203 Berlin
Germany
Phone: (+4930) 8104-1800
Fax: (+4930) 8104-1807
wolfgang.paatsch@bam.de

ITALY
Prof. Laura E. Depero
INSTM and Laboratorio di
Strutturistica Chimica
Università di Brescia, via
Branze 38
25123 Brescia
Italy
Tel: +39 030 3715472
Fax: +39 030 3702448
depero@ing.unibs.it

Dr. Eng. Anna Moreno
ENEA
Inn-Diff SP 59, Via
Anguillarese 301
00060 S Maria di Galeria
Rome
Italy
Tel: +39 6 3048 6474
Fax: +39 6 3048 4729
anna.moreno@casaccia.enea.it

Dr. Alessandro SANTORO
Operational Director UNI
Via Battistotti Sassi, 11b
20133 MILANO,
Italy
alessandro.santoro@uni.com

JAPAN
Dr Kiichi Oda
Chubu Collaboration Center
National Institute of
Advanced Industrial Science
and Technology
(AIST)
2266-98 Anaphora
Shimoshidami, Moriyama-ku
Nagoya 463-8560
JAPAN
Tel: +81-52-736-7057
Fax: +81-52-736-7403
k-oda@aist.go.jp

Mr. Toru Sato
Director
Office for Materials Research
and Development
Basic and Generic Research
Division
Research Promotion Bureau
Ministry of Education,
Culture, Sports, Science and
Technology (MEXT)
2-5-1, Marunouchi, Chiyoda-ku, Tokyo, 100-8959, Japan
Tel: +81-3-6734-4100
Fax: +81-3-6734-4102
tsato@mext.go.jp

Dr. Koichi Yagi
Supervising Researcher
National Institute for
Materials Science (NIMS)
1-2-1 Sengen, Tsukuba-shi
Ibaraki-ken 305-0047
Japan
Tel: +81 298 59 2803
Fax: +81 298 59 2801
yagi.koichi@nims.go.jp

UNITED KINGDOM
Mr. Michael H. Graham
Electrical International
Manager
BSI Standards
389 Chiswick High Road,
London W4 4AL,
UK
Tel: +44 181 996 7459
Fax: +44 181 996 7460
mike_graham@bsi-global.com

Dr. Kamal Hossain
Corporate Director,
National Physical Laboratory
Hampton Road, Teddington,
Middlesex TW11 0LW
UK
Tel: +44 20 8943 6024
Fax: +44 20 8943 6407
kamal.hossain@npl.co.uk

Dr. Graham Sims
Knowledge Leader,
Engineering and Process
Control Division,
National Physical Laboratory
Hampton Road, Teddington,
Middlesex TW11 0LW
UK
Tel: 00 44 (0) 20 8943 6564.
Fax: 00 44 (0) 20 8614 0433
graham.sims@npl.co.uk

LIAISON MEMBERS

Mr Mike Smith
Director, Standards
International Organization for
Standardization
1 rue de Varembe
1211 Geneva 20
SWITZERLAND
ISO Liaison
Tel. +41 22 7497280
Fax +41 22 7497349
smith@iso.ch

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