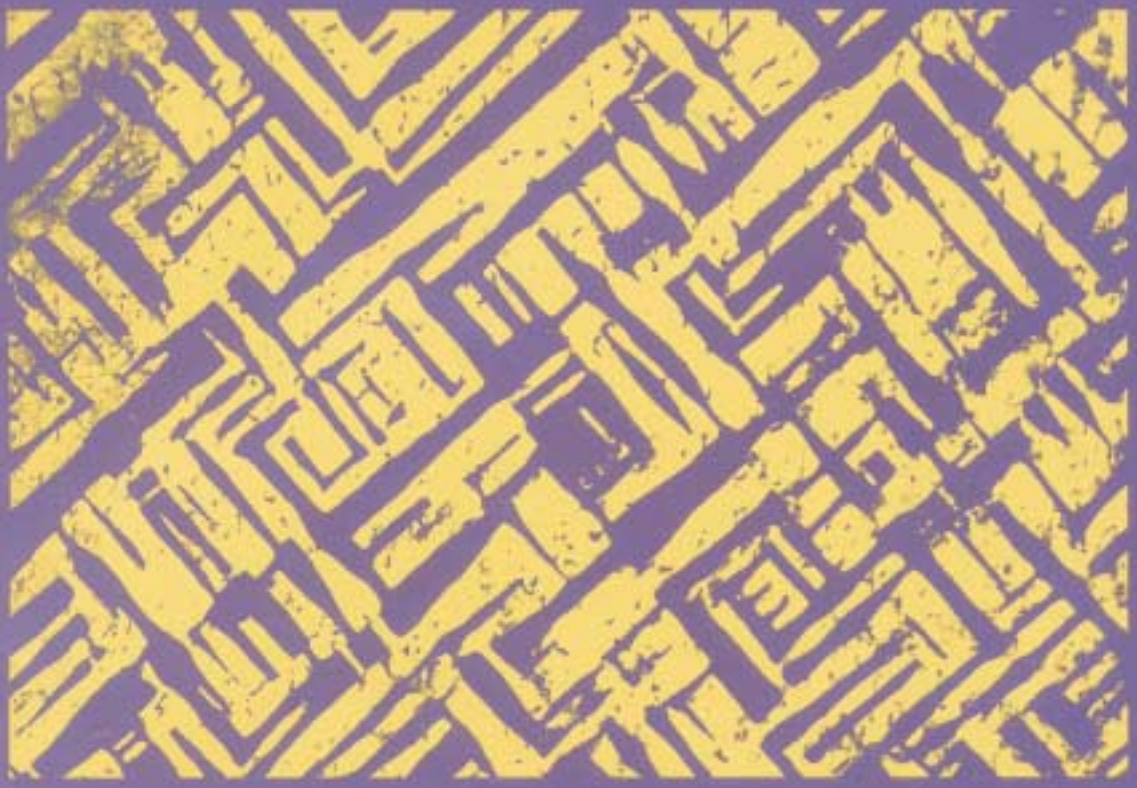
VAMAS



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

Website: www.vamas.org

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: Microstructure of titanium-aluminum-niobium (Ti-Al-Nb) alloy showing two-domain distribution of the orthorhombic **O** phase. The O phase is a candidate for use as a matrix in a new generation of high temperature composite materials for aerospace applications. The average spacing between domains is approximately 100 nanometers

Photograph courtesy of Leonid Bendersky, Metallurgy Division
Materials Science and Engineering Laboratory
National Institute of Standards and Technology
Technology Administration
U.S. Department of Commerce

VAMAS

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• Chairman's Message •

It is a pleasure to be able to report to you as Chairman of the VAMAS Steering Committee (SC). I am quite pleased with the progress that VAMAS has made during the past year. In April, we had a very successful SC meeting including the Technical Working Area's (TWAs) leadership in Gaithersburg, MD, held along with a joint SC/TWA Chair Workshop with the theme of VAMAS linkages to standards development organizations. As an introduction to this workshop, Mr. Charles Ludoph, the Deputy Assistant Secretary for Europe of the U.S. International Trade Administration, gave an insightful presentation on the "U.S. Perspective on the Eve of the WTO New Round of Trade Negotiations." Several VAMAS members, including representatives of standards development organizations (SDOs), also gave their views on linkages between SDOs and VAMAS, followed by presentations from each of the TWA chairs specific to their technical activities. During the Steering Committee meeting, special presentations were made by SC members from each VAMAS signatory country on critical issues facing their country. Summary minutes of the workshop and SC meeting can be found on the VAMAS web site (www.vamas.org).

I encourage anyone curious about VAMAS or interested in VAMAS activities to follow events through the VAMAS web site. The web site contains announcements of new projects and new Technical Working Area (TWA) topics, summary minutes of the most recent Steering Committee meeting, the current list of active VAMAS Projects, the list of standards developed all or in part from TWA inputs, the most recent compilation of TWA annual reports, and the latest issue of the VAMAS Bulletin.

There were several new initiatives agreed to at the Gaithersburg meeting. One of these was the approval of a new TWA on Quantitative Mass Spectroscopy of Synthetic Polymers, led by Dr. Charles Guttman of NIST. Also, a task group was created consisting of Dr. Leslie Smith (NIST), Dr. Horst Czichos (BAM), Dr. Marc Steen (JRC, EC), and Dr. Kamal Hossain (NPL), whose mission is to investigate a possible VAMAS role in Mutual Recognition Arrangements and closer ties to the International Bureau of Weights and Measures (BIPM). Finally, VAMAS will co-organize a workshop with CENSTAR in the general area of nanotechnology. The Workshop, June 5-6, 2002, will be co-chaired by Dr. Colin Lea of NPL and myself.

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

Steve Freiman
Chairman

• Feature Article •

NEW TECHNICAL WORKING AREA

TWA 28 Quantitative Mass Spectrometry of Synthetic Polymers

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INTRODUCTION

With recent advances in mass spectrometry it is possible to measure the molecular mass of some biological and synthetic polymers. Among the advances is a variation of time-of-flight mass spectrometry, MALDI ToF MS, in which laser ablation is used to produce charged polymers in the vapor state. This technique has the potential to be an absolute method for measuring the molecular mass distribution (MMD) of synthetic polymers, a long sought after goal in polymer characterization.

The mass distribution of a synthetic polymer is the most important molecular characteristic that impacts its processibility and macroscopic properties. At present there are no accepted absolute methods of determining the molecular mass distribution of synthetic polymers. Classical absolute methods yield only moments of the MMD. Some chromatographic methods, such as gel permeation chromatography, provide measures of the solution volume of the polymers. This then gives *relative* measures of MMD. Chromatographic instruments must be calibrated with polymer standards that have narrow MMD and are similar chemically and architecturally to the polymer being tested. These polymer standards are certified for *averages* of the molecular mass distribution by classical methods (light scattering, membrane osmometry, sedimentation equilibrium ultra centrifugation). Since such standards are unavailable for most polymers crude approximations are used which introduce uncertainties in the measurement results.

A new TWA has been established to develop mass spectrometry as a quantitative method to determine the molecular mass distribution, MMD, of synthetic polymers. The current principal goals are:

i. sample preparation techniques that minimize sampling bias in terms of molecular mass
ii. instrument settings that produce optimal signal-to-noise without discrimination in molecular mass distribution
iii. standards for calibration of mass spectrometers
iv. data analysis procedures that adequately account for factors that may distort molecular mass distributions and contribute to systematic uncertainty (Type B evaluation).

INDUSTRIAL BACKGROUND

Industrial needs using the MALDI method vary. Quantitation of both a homopolymer and a mixture of the same homopolymer but with different end groups is of importance at lower molecular masses. This is the range of molecular masses for polymers, which are used as reactive prepolymers.

The National Institute of Standards and Technology conducted an international interlaboratory comparison study of a polystyrene material by MALDI ToF MS. The Bundesanstalt für Materialforschung und –Prüfung (BAM) also conducted interlaboratory comparisons by MALDI ToF MS of polystyrene and other polymers for the purpose of evaluating classical methods.

A large number of industrial laboratories participated in these interlaboratory comparisons. In the NIST interlaboratory comparison of the 23 laboratories reporting results, 11 were industrial laboratories.

CURRENT STATUS AND OPPORTUNITIES

In the MALDI ToF MS method the sample consists of the analyte, a matrix material that absorbs the laser light and a metal salt to provide the charge to the analyte. The sample components are dissolved in a common solvent and deposited on a sample plate where, after solvent evaporation, a solid sample composed of analyte, matrix and metal salt remains. A UV laser pulse vaporizes the sample through absorption by the matrix substance producing a volatile mixture, including ions of the analyte that are accelerated by an electric field and analyzed by conventional ToF mass spectrometry.

The current status of MALDI ToF MS measurements on synthetic polymers is as follows:

1) Consistent results are obtained on synthetic polymers possessing polar groups, such as polyethylene oxide, and/or carbon-carbon double bonds, such as polystyrene or polybutadiene, of relatively low molecular mass and narrow MMD. The presence of polar groups, and/or carbon-carbon double bonds enhances the probability of charging; low mass leads to resolution at the monomer level and narrow MMD insures that detector saturation does not distort the measured distribution. However, disagreement exists between moments of the MMD calculated from mass data and values obtained by classical methods in most cases.

2) Currently, once the polymer mass exceeds about 500 x monomer mass, it is not possible to resolve polymer molecules differing in mass by one monomer group, but as long as the MMD is narrow meaningful results may still be obtained.

3) The technique fails to characterize polymers with broad distributions of molecular mass that is thought to arise from the detector being saturated by low mass species. (In ToF MS low mass species arrive at the detector first).

4) There is only one report of successful acquisition of MALDI ToF MS signals from polyethylenes with molecular masses up to about 5000 u. Analysis of higher molecular masses of polyethylenes and polypropylenes is still difficult. This is particularly troubling

since these two polymers dominate commercial markets and exhibit a rich variety of molecular architecture, depending on how they're produced, that gives them properties ranging from those of rubbers to those of engineering plastics. The commercial importance of these polymers will likely increase in the future owing to recent advances in catalysis which permit the control of molecular architecture, including the MMD, of these polymers to an unprecedented degree. A method of measuring MMD in these materials would allow researchers to establish the critical relationships among properties, molecular structure and synthesis needed to guide development of tailored-made materials.

5) In order for the MMD determined by MALDI ToF MS to be quantitative there must be no discrimination in volatilization/charging by molecular mass.

PLAN: QUANTITATIVE MMD

Research in the proposed TWA will build on knowledge and results gained in the interlaboratory comparisons of MALDI ToF MS sponsored by NIST and by BAM. A report on the NIST interlaboratory study has been published (Analytical Chemistry, Vol. 73, 1252-1262, 2001). The protocols from these studies will serve as the starting point for extending the method to polystyrene possessing different molecular mass distributions. In addition, collaborative work will be initiated in three distinct aspects of MALDI ToF MS: sample preparation, data acquisition and data analysis, including evaluation of Type B uncertainties. Progress in each aspect is needed to improve quantification of the method for MMD.

In the interlaboratory study mentioned above laboratories were asked to use a specific sample composition (matrix material, metal salt and solvent), but the method of depositing the sample on the target plate was not specified. The procedures currently used to prepare samples for MALDI ToF MS yield heterogeneous samples in which signal quality varies with the position of the focused laser beam. Thus, a common sampling procedure is to scan over the sample target until a 'sweet spot' is found from which a spectrum is observed. If the sample deposition method produces discrimination with respect to molecular mass the 'sweet spot' may not be a true representation of the MMD of the polymer. Ideally one would like to sample the polymer dissolved in a solvent to insure homogeneity and some progress in instrumentation towards liquid sampling has been reported. However, most commercial instruments in use have not been adapted to liquid samples, thus methods to improve sample homogeneity in the solid state are desirable. Electrospray has been used at NIST to produce more uniform samples and current activities include characterization of sample morphology to identify attributes that optimize the signal intensity and its quality. Recently researchers from the Max-Planck-Institute for Polymer Research in Mainz, Germany proposed a solvent-free method to prepare uniform matrix-analyte mixtures in MALDI and this too will be studied.

Participating laboratories would extend these studies to the full range of matrix materials and metal salts used in polystyrene analysis by MALDI ToF MS. An interlaboratory comparison will be conducted on the optimal sampling method to assess applicability with different commercial and home-built instruments.

It is known that fragmentation of the polystyrene in the plume produced by laser ablation or during transit to the detector may occur. Furthermore, adducts of the polystyrene with matrix molecule and metal atoms may also be present. In some cases these species

appear as secondary series of mass peaks in the spectrum. In either case they would affect the determined MMD. The power of the laser pulse is known to affect the degree of fragmentation. Comparison of linear and reflection modes of detection and ion selection methods are useful in revealing the presence of secondary species. A detailed investigation of these effects on molecular mass spectra from polystyrene with peak masses in the 4 ku and 7 ku ranges has been made at NIST. The proposed work would involve participating laboratories to extend these studies to higher mass polystyrenes, as well as to other types of commercial instruments. The purpose would be the identification of instrument critical parameters germane to all instruments and in cases where the settings are instrument specific to alert instrument operators to acquire data over a range of values of the parameters.

CONCLUSIONS

A number of methods to determine the MMD or its moments have been issued. ~~ASTM~~ Test methods are available for determination of MMD by chromatographic methods (ASTM D6474-99, ASTM D5296-97**,** DIN 55672-1, and DIN 55672-2) and the mass average molecular mass by light scattering (ASTM D4001-93). To our knowledge there are no organized activities within standards developing organizations to develop MALDI ToF MS as a quantitative method for determining the mass distributions of synthetic polymers. This TWA will focus on providing standard methodology for this new absolute method of MMD determination for synthetic polymers.

• Feature Article •

ASSURING MARKET RELEVANCE OF ISO TECHNICAL COMMITTEE ACTIVITIES – LINKING RESEARCH TO THE MARKET PLACE

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1 Changing times, changing needs, changing priorities

Over the last quarter century, the role of the International Organization for Standardization (ISO) has changed more than once, as the organization has striven to respond to the needs of its constituencies and stakeholders. No doubt further changes will be required in the future and the great challenge for all involved in ISO is not only to manage such changes, but also to integrate yesterday's priorities with those of today. At the same time, it must remain alert to the likely changes that the organization will need to undergo tomorrow.

To some, it may seem odd to hear a professional standardizer talking about change. The standards organizations, or rather the formal standardization system in general, are often reported to be conservative, slow and bureaucratic, hardly the terms one would use to describe an organization that embraces change. However, a culture of change is one that has become indispensable to any organization trying to respond to the dynamics of technological innovation, globalization of markets and political evolutions at the dawn of the twenty first century.

It is certainly true that, following its creation in 1947, ISO was for the first quarter century of its existence pretty much a backwater in terms of its significance. This is not too surprising as this was the period of national reconstruction following WW2; national interest was still of paramount importance and national standards were still essentially the tools used to regulate markets which were essentially domestic. ISO's role during this period was largely to develop recommendations to aid its members to harmonize their national standards, although it is doubtful that many national standards during that period were based on ISO publications that at that time were issued as ISO Recommendations.

Following the decision in the early 1970s that the results of ISO's work should be published as ISO International Standards, by the 1980s, it was evident that an explosion was taking place in international trade and for the first time ISO found itself in the position of having to develop standards on new subjects instead of harmonizing national standards as in the past. This also brought with it the challenge to work rapidly. Until that time,

standardization was often used as a tool to consolidate a market place based on a good few years experience with products in the market. The new requirement was to develop standards that would help to stabilize emerging markets and the availability of standards at the right time therefore became increasingly important.

By the end of the 1980s, the growth in international trade seemed essentially to have secured ISO's future and it was therefore natural that, in the lights of trends at that time, when ISO looked to expand its relevance, it turned towards the world of pre-standards and pre-standardization research.

2 The Advisory Board on Technological Trends (ABTT)

The ABTT was established in 1987 comprising a number of high-level individuals from industry and academia essentially to brainstorm and advise ISO and IEC on the trends they saw in industry, how they expected technologies to evolve and the likely consequences in terms of needs for international standardization.

In their report, they noted that in particular industries (electronics, information technology, aerospace and synthetic materials), significant changes had occurred over the previous two decades and that the process of innovation, comprising scientific discovery, applied research and development, production and commercialization, had essentially evolved to become a series of simultaneous and interactive processes. Looking ahead, and remember that at that time few people had heard of the Internet or the World Wide Web, they foresaw the convergence of information technology and telecommunications, with consequently a broad dissemination of engineering, production and control practices leading to transnational production lines. They considered that this globalization of industries and markets would lead to international standardization increasingly taking precedence over national and regional standardization because only international solutions could meet the needs of geographically dispersed and vertically integrated industries.

Of more immediate relevance, they recognized that in the new interactive process of innovation, islands of standardization emerged at various steps in the overall process. For example, during the phase of scientific discovery, terms and definitions needed to be established to describe new phenomena and concepts and it is a generally held view that in any standardization process the first agreements need to be reached on a common understanding of terms and their definitions. In the next phase, i.e. applied research and development, the general requirement was to be able to characterize new products, which requires the standardization of appropriate methods of test. And finally, in order to ensure that innovation is not stifled, they confirmed ISO's general policy that product standards should be written in performance, rather than descriptive, terms.

With this as background, ISO looked to consolidate its links with the world of pre-standardization research and it was in this context that ISO's cooperation with VAMAS was established and a new type of publication, called Technology Trends Assessments (TTA), was established. Although other ways of producing TTAs were envisaged, these have never materialized and VAMAS remains the sole originator of TTAs within the ISO system.

It should not be inferred from this that ISO has not otherwise established links with the

research world. Such links indeed exist but, more often than not, the results of such work are fed directly into the formal standardization process without passing through the intermediate step of publishing a TTA.

3 The Vienna Agreement, the consortia threat and market relevance

If the 1980s can be looked back upon as a period of expansion and optimism, the 1990s proved to be one during which ISO had to adapt itself to a number of evolutions that threatened the relevance of the international standardization system. The first of these evolutions concerned the political decision taken in the late 1980s to create the Single European Market, based upon a "new approach" to legislation within the European Union which required that formal legislation be defined in general terms as "essential requirements" intended to protect public health and safety and the environment, supported by a portfolio of voluntary, consensus European Standards developed by the European Standards Bodies. Much has been written on this subject and it will suffice to say here that, based on the premise that the Single European Market needed to be integrated into the global market, then there was recognition that the standards which regulated the SEM should also be those which regulated the market at the global level. This consequently resulted in the establishment of a cooperation agreement between ISO and its European counterpart (CEN), known as the Vienna Agreement, which lays out processes for collaboration including the procedures for the development simultaneously of International and European Standards. The agreement has served both organizations well and has helped to dispel the fears that the European Union was about to construct "fortress Europe".

The second evolution that caused concern was the emergence in some industry sectors of industrial consortia and fora as preferred mechanisms for developing agreements within industry on standards, in preference to the development of such standards in the formal standardization system. Initially limited to the information technology sector, consortia have emerged in other areas but are generally limited to rapidly moving technologies. A good deal of time was spent trying to understand this development, and whilst the conclusions were fairly complex, the underlying message was that industry did not in all cases need to rely on fully developed International Standards representing the broad consensus required for an ISO International Standard.

Recognizing that during the development of International Standards, three successive levels of consensus are attained, ISO decided to allow committees the option of publishing either standards, representing the highest level of consensus, or intermediate publications representing lower levels of consensus. Hence, once agreement is reached between experts in an ISO working group, they now have the option of progressing the document further as a committee draft or requesting that it be published as an ISO Publicly Available Specification. Similarly, once at the committee stage a consensus has been reached between national positions, committees now have the option of progressing the document further as a draft International Standard or requesting its publication as a Technical Specification. As a separate development, the ISO Council also agreed to introduce the possibility of developing documents outside the normal ISO committee processes in workshops, the output being published as "Industry Technical Agreements".

It was considered that these new products would place ISO in a better position to respond to real needs of market players. However, at the same time, some were questioning how

useful some ISO standards were and others were claiming that they no longer represented the state of the art.

4 ISO 9000 and the management systems backlash

I will come back to what ISO has been doing to assure the market relevance of its work but to illustrate some of the dynamics I would like to relate the case of ISO 9000.

When ISO 9000 was first published in 1987, many expected it to be popular but not to the extent that occurred. Indeed the authors had originally foreseen the standard being used primarily in first and second party situations, i.e. self-declaration and supplier-client situations. What in fact happened was that the market place in many instances required third party certification and in some instances, particularly in government procurement, an ISO 9000 certificate became indispensable if one wished to remain in business.

Although work on environmental management systems was successfully completed in 1996, the last few years have seen a mobilization within industry, strongly opposed to ISO developing any other type of management system standard. They claim that such standards cause unnecessary costs and add no value to organizations. As a consequence, requests that ISO address privacy and protection of personal data, occupational health and safety management systems and risk management have all been rejected. Perhaps this speaks well for ISO's democratic processes. However, the truth of the matter is that such standards are needed as has been demonstrated in particular by the Australian risk management standard that has become a worldwide best seller. The real problem I would suggest is the fear that the market place might make a particular voluntary standard again essential to remaining in business and some are not willing to take the risk. In consequence, the only option they support is no ISO standard on such "dangerous" subjects.

The ISO Central Secretariat was itself certified to ISO 9002 several years ago and I would have to say that from my perspective I believe it was a very worthwhile exercise because it helped us to "standardize" management disciplines across the organization. It also helped us to think about how we were organized, our relationships with our different suppliers and customers and basically gave us an overall methodology. I would add that we did this because we wanted to and not because anyone was requiring it of us and that in some instances we are now on paths that take us well beyond the minimum requirements of ISO 9000.

5 Three cornerstones for market relevance

The question of the market relevance of ISO's technical work has been the first priority on ISO's strategic agenda for the last few years and three different approaches have been introduced to try to ensure that when ISO undertakes work it is because there is a real need for it, and that that need is periodically reconfirmed.

The first approach has been a requirement that each ISO committee prepare a business plan, agreed between the committee members and subject to review and approval by the Technical Management Board. Such business plans contain much in the way of factual data (structure of the committee, membership, work programme) but the most important element I believe is that in which the committees have to agree on the market needs that

they are trying to address, how they see the market evolving and how they see the priorities. Such business plans are being made freely accessible on a public access website so that anyone, anywhere can comment on the assumptions, priorities etc. at any time. This will help to install a dialogue between market players not usually represented in the standards system and those that are represented and should help to give much better assurance that what is being done responds to market needs.

The second of these approaches has seen the introduction of what is called a "standards value assessment tool"(SVAT). Simply put, this is a matrix by which those responding to questions such as: do you think a standard on this subject is needed? can identify what they believe such a standard would contribute to, for example, public health and safety, industrial efficiency, and international trade. These responses are to be aggregated at the national level to determine a national response and then at the international level to determine whether the standards project should be undertaken, what the benefits are expected to be and how important the project is (i.e. its priority relative to other projects).

The third approach has been a policing by the Technical Management Board of committee work programmes. This latter decision requires the ISO Central Secretariat to cancel automatically projects on which no progress has been made for at least three years and also to cancel projects that have not reached the final stages after seven years. In order to continue such work, committees are required to re-allot the project proposals to determine that there is a still a market need and this will be even more evident with the new SVAT.

The result of this latter action in particular has seen a reduction of the total ISO work programme from 7000 projects to some 4800 at the present time and with it a stronger assurance that the standards being published are indeed really needed by the market place.

6 Closing the loop

This brings me back, to conclude, to ISO's collaboration with VAMAS. Although we are at an early stage in our business planning activity, and it will take some time before the dialogue between market players and ISO committees is well established, one might reasonably expect that some of the emerging requirements identified by market players might be appropriate topics for VAMAS TWAs to undertake. I would therefore invite TWA chairs and others active in VAMAS to review periodically the business plans of ISO/TCs to map their own relevant activities to perceived market needs. Thus, VAMAS will also be well placed to demonstrate the market relevance of the work it is undertaking.

• Feature Article •

VAMAS CONTRIBUTIONS TO STANDARDS DEVELOPMENT

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It has become apparent in recent years that VAMAS should strengthen its relationships with Standards Development Organizations (SDOs) because they are the primary clients for VAMAS outputs. The timeliness of this issue became very clear during the 1999-2000 Steering Committee (SC)/Technical Working Area (TWA) discussions leading to the development and implementation of the VAMAS Strategic Plan. A tentative decision had already been made previously to schedule a joint SC/TWA workshop meeting as part of the 2001 regular SC meeting. After a review of possible topics, **"VAMAS Linkages to Standards Development Organizations (SDOs)"** was selected as the workshop focus.

The workshop was held 9-10 April 2001, just prior to the 26th SC meeting at the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland, USA. The opening session included a series of invited introductory presentations on standards issues. Speakers included the Deputy Assistant Secretary for Europe, U.S. International Trade Administration, the ISO liaison to VAMAS, the Secretary of the UK Electrotechnical Committee and SC member, and the past chairman of a VAMAS TWA. The following session contained individual presentations from each TWA describing the SDO linkages to the TWA, including success stories and problems. In addition, each TWA reviewed the national, regional, or international standards developed (draft, revised, or new), standard reference materials, and Technology Trends Assessment (TTA) documents that are based all or in-part on work and outputs of the TWA.

Based on information gathered from the 2000-2001 TWA Annual Reports, the TWA workshop presentations, and SDO web sites, the Secretariat has compiled an initial inventory of draft standards, revised standards, new standards, standard reference materials, and Technology Trends Assessment documents developed all or in-part based on technical contributions from VAMAS TWAs. Currently, VAMAS has contributed to 64 standards at various stages of development, including: 30 ISO standards, 8 IEC standards, 11 CEN standards, 9 ASTM standards, 3 JISC standards, and 3 BSI standards. In addition, there have been three Standard Reference Materials developed or under development and four Technology Trends Assessment reports published. The following table summarizes a portion of the inventory.

Table 1. List of draft standards, revised standards, new standards, standard reference materials and Technology Trends Assessment publications based all or in part on technical contributions from VAMAS Technical Working Areas (TWAs).

[In the table, the first column identifies the Standards Development Organization (SDO) or other organization, the second column identifies the document number, and the third column identifies the subject of the document.]

STANDARDS

TWA 1	**Wear Test Methods**	
CEN	ENV 1071-6	Ball cratering wear testing
TWA 2	**Surface Chemical Analysis**	
ISO	ISO 14606	Sputter depth profiling using layered systems
ISO	ISO 14975	Information formats
ISO	ISO 14976	Data transfer format
ISO	ISO 15472	Energy scales (XPS)
ISO	DIS 17973	Energy scales for elemental analysis (AES)
ISO	DIS 17974	Energy scales for elemental and chemical state analyses (AES)
ISO	CD 19318	Methods used for charge control & correction (XPS)
ISO	WD 18327	Unintended degradation by XPS
ISO	AWI 18118	Relative sensitivity factors for quantitative analysis of homogeneous material (AES, XPS)
ISO	TR 15969	Sputtered depth
ISO	DTR 16268	Reference material for ion-implanted surface
ASTM	E 1636	Sputter depth profile interface data
ASTM	E 2108	Electron binding-energy scale of x-ray photoelectron spectrometer
TWA 3	**Ceramics for Structural Applications**	
ISO	ISO 14705	Hardness of monolithic ceramics at high temperature
ISO	ISO 18756	Fracture toughness at room temperature by the surface crack in flexure (SCF) method
ISO	DIS 15732	Fracture toughness of monolithic ceramics at room temperature by single edge precracked beam (SEPB) method
ISO	CD 17565	Flexural strength at elevated temperature
ISO	NWI xxxx	Chevron notched beam fracture toughness method
CEN	ENV 623-1, Part 1	Grain size measurement
CEN	ENV 623-5, Part 5	Phase volume fraction
CEN	ENV xxxx, Part 2	Room temperature fracture toughness with single edge precracked beam (SEPB) method
CEN	ENV 843-3, Part 3	Subcritical crack growth from constant stressing rate flexural tests
CEN	ENV 843-4, Part 4	Vickers, Knoop and Rockwell superficial hardness tests
CEN	ENV 843-6, Part 6	Guidelines for fractographic investigation
CEN	ENV xxxx, Part 4	Fracture toughness at room temperature by the surface crack in flexure (SCF) method
CEN	ENV xxxx, Part 5	High temperature fracture toughness by the single edge precracked beam (SEVNB) method

ASTM	C 1322	Fractography and characterization of fracture origins in advanced ceramics
ASTM	C 1326	Knoop indentation hardness of advanced ceramics
ASTM	C 1327	Vickers indentation hardness of advanced ceramics
ASTM	C 1421	Fracture toughness of advanced ceramics at ambient temperatures
JISC	JIS R 1607	Fracture toughness of high-performance ceramics
JISC	JIS R 1617	Fracture toughness of fine ceramics at elevated temperature

TWA 5 Polymer Composites

ISO	ISO 15024	Mode I Interlaminar fracture toughness for unidirectionally reinforced materials
ISO	DIS 13003	Fatigue properties under cyclic loading
ISO	xxxx	Mode II fracture toughness

TWA 13 Low Cycle Fatigue

ISO	ISO 1099	Axial load fatigue testing
ISO	ISO 12111	Thermal-mechanical fatigue testing
ISO	ISO xxxx	Machine alignment for axial fatigue testing
ISO	DIS 12106	Axial strain-controlled method
BSI	BS 7270	Constant amplitude strain-controlled fatigue testing
JISC	JIS Z 2279	High temperature low cycle fatigue testing

TWA 14 United Classification System for Advanced Ceramics

ISO	ISO 15165	Classification system for fine ceramics
CEN	12112	United method for classification
ASTM	C 1286	Classification for advanced ceramics

TWA 16 Superconducting Materials

IEC	IEC 61788-2	DC critical current of NB_3Sn composite superconductors
IEC	IEC 61788-3	DC critical Current of Bi-2212 and Bi-2223 oxide superconductors
IEC	IEC 60050-815	International electrotechnical vocabulary (Part 815) – superconductivity
IEC	FDIS 61788-7	Surface resistance in thin film superconductors at microwave frequencies
IEC	CDV 61788-8	AC losses in superconducting wires (NbTi) – pick-up-coil method
IEC	CDV 61788-10	Critical temperature of composite superconductors
IEC	CDV 61788-13	AC losses in superconducting wires (NiTi) – megnetometer method
IEC	CDV 61788-14	Trapped flux density in large grain oxide superconductors

TWA 17 Cryogenic Structural Materials

ISO	AWI 19819	Tensile testing in liquid helium

TWA 21 Mechanical Measurements for Hardmetals

ISO	DIS 3327	Transverse rupture strength

TWA 22 Mechanical Property Measurements of Thin Films

and Coatings

ISO	DIS 14577-1.2	Test Method for indentation testing
ISO	DIS 14577-2.2	Verification and testing machines for indentation testing
ISO	DIS 14577-3.2	Calibration of reference blocks for indentation testing
CEN	ENV WI 152	Determination of hardness and Young's modulus by instrumented indentation testing

TWA 25 Creep/Fatigue Crack Growth in Components

BSI	PD 6539	Influence of crack growth on significance of defects in components at high temperature
BSI	BS 7910	Acceptability of flaws in metallic structures
ASTM	E 1457-92	Creep crack growth in ductile materials
ASTM	E 1457-98	Creep crack growth in more brittle materials

Note: Items for which the SDO has not yet assigned a document number are identified by **xxxx**

STANDARD REFERENCE MATERIALS

TWA 3 Ceramics for Structural Applications

NIST	SRM 2100	Fracture Toughness of Ceramic
NIST	SRM 2830	Knoop Hardness
NIST	SRM 2831	Vickers Hardness (under development)

TECHNOLOGY TRENDS ASSESSMENT PUBLICATIONS

TWA 14 United Classification System for Advanced Ceramics

ISO	ISO TTA 1	Unified Classification System

TWA 15 Metal Matrix Composites

ISO	ISO TTA 2	Tensile Tests for Discontinuously Reinforced Metal Matrix Composites at Ambient Temperatures

TWA 16 Superconducting Materials

IEC	IEC TTA 2	Critical Current Measurement Method for Nb_3Sn Multifilamentary Composite Superconductors

TWA 20 Measurement of Residual Stress

ISO	ISO TTA 3	Measurement of Residual Stress by Neutron Diffraction

Standards Development Organizations (SDOs)/Other Organizations

ISO International Organization for Standardization
IEC International Electrotechnical Commission
CEN European Committee for Standardization
ASTM American Society for Testing and Materials
JISC Japanese Industrial Standards Committee

BSI British Standards Institution
NIST National Institute of Standards and Technology

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 40
"A Round Robin to Measure the Thermal Conductivity
of Silicon Dioxide Films"
by
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A round-robin to measure the thermal conductivity of thin films was carried out by VAMAS Technical Working Area 23, Thermal Properties of Thin Ceramic Films, based on a recommendation from the Workshop of Thin Film Thermal Conductivity [1,2] held at the Thirteenth Symposium on Thermophysical Properties, June *25-26,* 1997. Thin film thermal conductivity is one of the parameters needed to predict the performance of electronic devices. Of particular interest is the thermal conductivity of insulating layers, such as silicon dioxide, that might impede the flow of heat generated in active circuit elements to heat sinks. Currently there are no standard methods of measuring thin film thermal conductivity. Thus, it was concluded that a round-robin to measure the thermal conductivity of silicon dioxide films on silicon produced by oxidation of silicon wafers was appropriate.

Experience had shown that silicon dioxide films produced by oxidation of silicon were stable and could be made reproducibly. Specimens with nominal film thicknesses of 50 nm, 100 nm, 200 nm, and 500 nm were prepared by oxidation of silicon wafers. The thicknesses of the films were determined by means of scanning ellipsometry. The 50 nm, 100 nm, 200 nm silicon dioxide films were obtained by oxidation of silicon wafers in dry oxygen at 950 °C. The 500 nm silicon dioxide films were prepared by oxidation of silicon wafers in steam at 1050 °C.

Several wafers of each thickness were produced in a single charge in order to have a sufficient amount of material for distribution to the participants. Each laboratory chose its own measurement methods. Eight different methods were used and results were obtained from 11 laboratories. However, six of the participating laboratories used the three-omega (3-ω) method which was the only method that was used by multiple laboratories. Thus, a complete interlaboratory comparison was carried out only for the 3-ω method.

The 3-ω method has been used extensively to measure the thermal conductivities of bulk and thin film dielectric materials. The method employs a metallic strip in intimate contact with the specimen surface. An AC electrical current is induced to flow in the strip causing heat generation in the strip. The heating has both a DC component that changes the average temperature of the specimen and an AC component that generates thermal waves in the specimen. The technique actually determines the total thermal resistance between the deposited metallic strip and the specimen substrate. Thus, when computing the conductivity, the interfacial thermal resistance may have to be taken into account.

Based on the results, it was concluded that the 3-ω method could form the basis of a standard measurement method. The 3-ω method showed very good inter-laboratory reproducibility. The average thermal conductivity of $Si0_2$ from the 3-ω data agrees very well with the handbook (bulk) value. If interface thermal resistance, R_o, is ignored, then the thermal conductivity calculated from the data appears to decrease with decreasing film thickness. However, if we assume that the interface thermal resistance is finite and that the thermal conductivity and the interface thermal resistance are specimen independent, the conductivity of $Si0_2$ =1.38±0.05 $W \cdot m^{-1}K^{-1}$ at 20 °C which is almost equal to the handbook value 1.37 $W \cdot m^{-1}K^{-1}$; R_o=(23±9)x1O^{-9} $m^2K \cdot W^{-1}$. The standard uncertainty of the mean value is the 95 % confidence limit. Multiple specimens having different film thicknesses can be used to calculate the interface thermal resistance if the thermal conductivity is independent of thickness. However, the formation of the metallic strip on the specimen surface requires a fabrication method, such as microlithography, that produces a highly adherent strip of precisely controlled dimensions. Such facilities may not be readily available to implement this standard.

Conclusions for the 3-ω method are: 1) inter-laboratory reproducibility is good; 2) uncertainty increases with decreasing film thickness; 3) interface thermal resistance cannot be ignored unless it is negligible compared to the thermal resistance of the film; 4) the special techniques required for specimen preparation make the procedure difficult to implement as a standard measurement method. 5) these conclusions are applicable only to the specimen-type used here (i.e. SiO_2, on Si); use of other specimen-types would require further validation.

With the successful conclusion of this round robin and the preparation and distribution of this final report, TWA 23 formally ended its activities. A draft document has been prepared and will be submitted to ISO as a proposed Technology Trends Assessment

References

1. Conference Report, “Workshop on Thin Film Thermal Conductivity Measurement at the Thirteenth Symposium on Thermophysical Properties, Boulder CO, June 25-26, 1997” A. Feldman and N. Baizaretti, J. of Res. NIST 103, 107-116 (1998).

2. “Review of a Workshop on Thin Film Thermal Conductivity Measurements”, A. Feldman, N.M. Balzaretti, A.H. Guenther in *Laser Damage in Optical Materials: 199”,* G.J. Exarhos, A.H. Guenther, M.R. Kozlowski, and M.J. Soileau, Editors, Proc. SPIE Vol. 3244, pp. 420-433 (1998).

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 41
"Quantifying Data Uncertainties and the Validation of a Code of Practice for the Measurement of Bending in Uniaxial Fatigue Test Pieces"
by
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This report highlights the main outputs from a recent international collaborative study that was partly funded by the European Commission. The project involved 8 partners and 20 associated partners from 12 countries. These included 11 from Europe and Japan, which participated through VAMAS TWA 13, Low Cycle Fatigue.

Details are presented of inter-comparison low cycle fatigue tests at ambient and at high temperatures. The study also included extensive measurements of specimen bending due to non-alignment of the load-train components in the test machines. A number of high temperature fatigue tests were performed with deliberately introduced specimen bending. The findings from all the above were then used to develop two procedures, one for the verification of alignment of uniaxial testing machines and the other for quantifying uncertainties in low cycle fatigue lifetime data.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS/ISO Technology Trends Assessment, ISO/TTA 3
"Measurement of Residual Stress by Neutron Diffraction"

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and
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Neutron diffraction is a technique that can be applied for determining residual (and applied) stresses in crystalline materials. With the method, elastic strain is measured and stress calculated using the elastic properties of the material. The depth to which these measurements can be made non-destructively within specimens or components depends on their size and shape. It is also dependent on the neutron scattering and absorption characteristics of the materials of which they are made. Typically the depths to which these measurements can be obtained are up to a few centimeters in most materials of practical interest.

No standard or code of practice is available for making residual stress measurements by neutron diffraction. As a consequence VAMAS TWA 20 was set up in January 1996 with the aim of carrying out the under-pinning research necessary for preparing a standard. The specific objectives of TWA 20 were to:

- establish accurate and reliable procedures for making non-destructive residual stress measurements by neutron diffraction,
- examine a selection of samples in which residual stresses had been introduced by different techniques,
- conduct inter-laboratory comparisons to establish reproducibility,
- assemble the necessary information for preparing a draft standard for making the measurements.

A European (EU) project RESTAND (Residual Stress Standard using Neutron Diffraction) was also started in December 1997 to demonstrate the application of the technique to industrial situations. This ISO/TTA presents the findings of these two investigations and contains the draft protocol for making the measurements until a standard has been developed. It includes the scope of the method, an outline of the technique, the calibration and measurement procedures recommended, and details of how the strain data should be analyzed to calculate stresses and establish the reliability of the results obtained. Relevant

committees concerned with the preparation of this standard are CEN/TC 138 AHG 7, and ISO/TC 135/SC 5 and ASTM E 28.13.

Both the VAMAS TWA 20 and RESTAND investigations involved a series of round-robin experiments. These were carried out by making measurements on the same samples at a number of neutron sources. Most of the sources world-wide that are capable of making the measurements participated. For the VAMAS TWA 20 project, four types of samples were examined. These were a shrink-fit aluminum alloy ring and plug, a ceramic matrix composite, a nickel alloy shot-peened plate and a ferritic steel weldment. These examples were chosen to establish the range of application of the technique. In each case a protocol was specified which each participating group was required to follow. Measurements were made at each neutron source independently. The results were then collected together and statistical analyses carried out to determine the reliability of the measurements. Data were collected on steady state instruments that used a monochromatic beam of neutrons and also on time-of-flight instruments that employed a pulsed polychromatic beam. With a monochromatic source, measurements are made on specific crystallographic planes; with the time-of-flight method the entire spectrum can be analyzed using profile refinement to obtain strains. Comparable results are obtained from each type of instrument.

The ring and plug assembly was the first specimen to be measured because a discontinuity in residual stresses is obtained at the ring/plug interface and comparisons can be made with theory. The ceramic matrix composite was chosen to determine the feasibility of making measurements in a dual phase system containing fine fibers. The shot-peened plate was selected to establish whether steep stress gradients (of the order of 2000 MPa/mm) could be measured close to external surfaces and the ferritic weldment to determine whether reliable results can be obtained through regions of different microstructure (and possibly chemical composition).

The studies on the ring and plug assembly established the basic procedures that should be followed [1]. It has been found that it is essential to ensure accurate positioning and alignment of a specimen in the neutron beam for reliable results to be obtained. A suitable shape and size of ‘gauge volume’ over which individual measurements should be made to achieve adequate resolution in regions of strain gradients has been identified. From statistical analysis of the data, it has been established in most cases, that a positional accuracy with a standard deviation of 0.1 mm can be achieved provided proper alignment procedures are adopted. It has also been ascertained that strains can be recorded away from surfaces to a tolerance of $\pm 10^{-4}$ corresponding to a stress of ± 7 to ± 20 MPa in most materials. Close to surfaces (or interfaces) and regions of variable microstructure, greater errors can be expected. Where the volume from which neutrons are being counted is traversed through a surface, there is the possibility that compensation is needed for the change in shape and size of the volume of material being sampled affecting the position at which the strain measured should be recorded.

A main aim of the RESTAND project was to develop industrial confidence in the application of the neutron diffraction technique for residual stress measurement. Measurements have been made on felt and fiber-reinforced composites for heat insulation and thermal shock resistance, on deep-rolled crankshafts to represent complex shapes, a quenched component and through fusion, linear-friction and friction-stir welds for power generation and aerospace applications. For composites, with fiber and matrices of similar

composition, it has been found that it is sometimes necessary to separate out the effects of overlapping peaks. With complex shapes such as crankshafts, care is needed to avoid orientations that involve long neutron path lengths to minimize attenuation.

Similarly curved surfaces can exaggerate surface aberrations. In all cases, it has been determined when using a monochromatic beam of neutrons, that measurements should be restricted to those planes which give high peaks close to a diffraction angle of 90° and which represent bulk material behavior.

[1] Webster, G.A., (Ed), 'Neutron diffraction measurements of residual stress in a shrink-fit ring and plug', VAMAS Report No. 38, January 2000.

Technical Working Area 1

WEAR TEST METHODS

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The major activity since the last report has been to gather ideas for the development of a procedure for the calibration of force sensor in the Nano-Newton range. Such low forces are encountered in Atomic Force Microscopy (AFM), which is increasingly applied for friction measurements in micro- and nanosystems in nanotribology. Until now the forces measured with an AFM are calculated from the force (or spring) constant of the Si-cantilever reported by the manufacturer and multiplying that by the measured cantilever deflection due to normal and friction forces acting on the tip of the cantilever. The spring constants of such cantilevers are calculated from the Young's modulus of the cantilever material and the dimensions of the cantilever. But the relative uncertainty of the dimensions lead to a high uncertainty of the force constant and a large deviation between the reported and real constants.

Status of current Projects

At present there are three active projects. The objective of Project No. 1, Compilation of Wear Test Standards, is to provide a validated source for standardized wear test methods that can be used by engineers and tribologists. The preparatory work for this is now complete. An NPL report containing a compact disk with the database [NPL Report MATC(A)07, September 2001] is now available, and has been used by one UK participant to identify wear-testing standards in an unfamiliar area. There has already been enthusiastic response from UK experts, and a US participant has also found the database useful.

The goal of Project No. 2, Ball Cratering Wear Testing is to establish this method as a standard and to produce a recommended test procedure. The draft test procedure for this has been written and potential participants identified in the USA and Europe. The draft procedure has already been written as a draft CEN. Funding has been gained from the EU to support the standardization of this test method. A preliminary interlaboratory exercise will take place in the next year to assess the validity of the draft standard.

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

Technical Working Area 2

SURFACE CHEMICAL ANALYSIS

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Since its inception, TWA 2 has initiated 41 TWA projects. Twenty-four projects have been completed and there are three inactive projects that could not be completed for lack of resources. The 14 current active TWA projects are listed below.

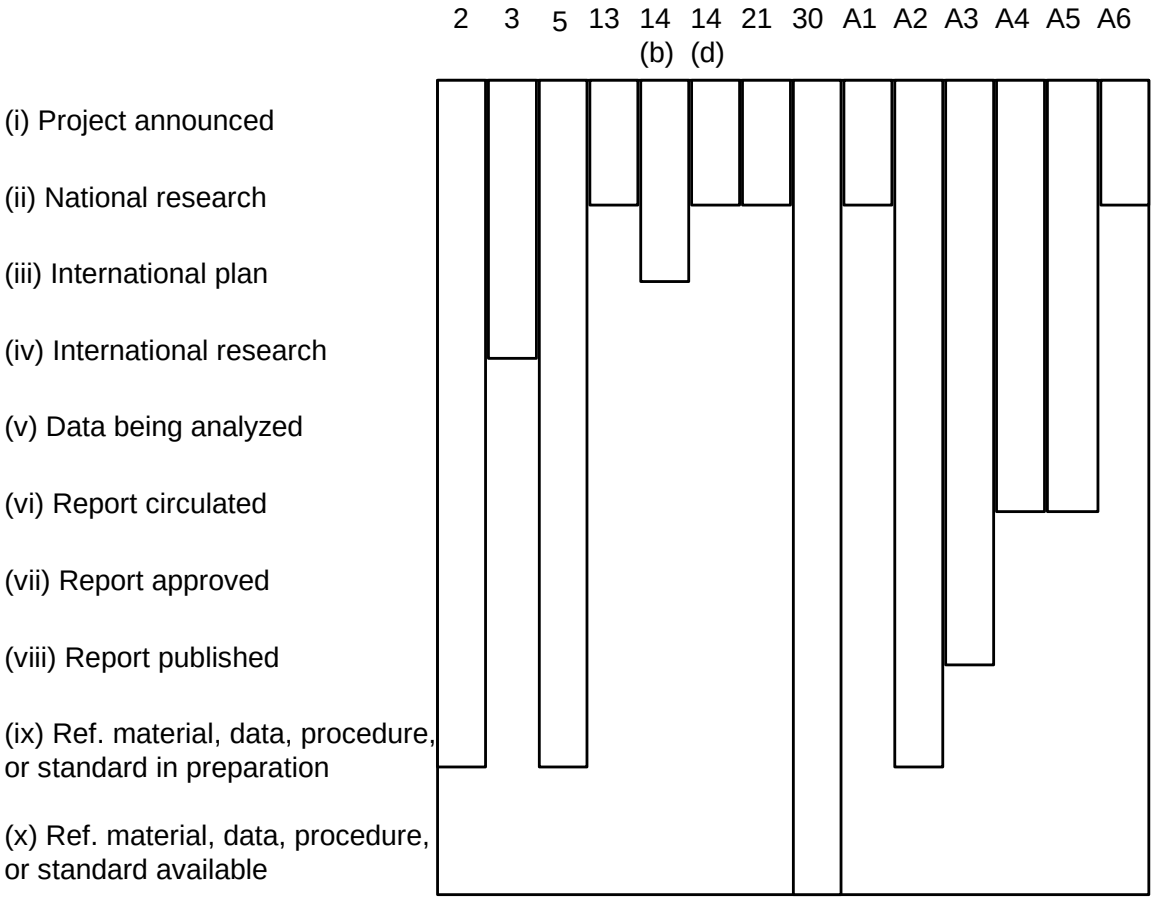
No.	Project Title
2.*	Development of calibration data for the energy scales of Auger-electron spectrometers
3.	Procedures for quantitative X-ray photoelectron spectroscopy
5.*	Development of reference materials prepared by ion implantation
13.	Tests of algorithms for data processing in AES - Factor analysis and intensity
14.*	(b) Tests of algorithms for background subtraction in AES (d) Tests of algorithms for angle-resolved XPS
21.	Tests of algorithms for the analysis of multicomponent spectra in XPS
30.	Development of a Common Data Processing System for AES and XPS
A1.	Use of the infinite velocity method for SIMS quantification
A2.	The evaluation of static charge stabilisation and determination methods in XPS on non-conducting samples
A3.	Interlaboratory study of static SIMS repeatability and reproducibility
A4.	Evaluation of multilayer reference coatings for quantitative GDOES depth profiling
A5.	Interlaboratory study of the degradation of organic materials in XPS analysis
A6.	Evaluation of uncertainties in XPS peak intensities associated with different techniques and procedures for background subtraction

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

The states leading active TWA 2 projects are as follows:

Canada	A1	UK	2, 21, A3
Germany	5, 13, A2, A4	USA	2, 3, A6
Japan	30, A5	EC	14(b), 14(d)

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



Many of the outputs of TWA 2 projects have been incorporated or are in the process of being incorporated into ISO standards. Table 1 identifies the standards or draft standards of ISO Technical Committee 201, Surface Chemical Analysis, arising from or related to TWA 2 projects. TWA 2 is a category-A liaison body with ISO/TC 201 and its subcommittees, and there is excellent communication between these groups.

There has been satisfactory progress in the active TWA 2 projects during the past year. Four interlaboratory comparisons have been completed (projects A2, A3, A4, and A5) for which there were 26, 18, 16, and 41 participants, respectively.

Project 30 has been brought to completion during the past year. This project involved the development of the “Common Data Processing System” (COMPRO) by Dr. Kazuhiro Yoshihara of the National Research Institute for Metals in Japan (this Institute will become the National Institute for Materials Science on April 1). COMPRO is software designed to facilitate the sharing of surface-analytical spectral data and the use of certain data-processing software. Spectra can be converted from the ASCII format to the VAMAS/ISO format (project 10), and procedures such as deconvolution, background subtraction, differentiation, quantitative analysis, and

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

ISO/TC 201 Subcommittee	Title of standard or draft standard
SC2: General Procedures	DTR 16268: Ion-implanted surface-analytical reference materials
SC3: Data Management and Treatment	ISO 14976: Data transfer format ISO 14975: Information formats WD 15760: Data dictionary for of X-ray photoelectron and Auger electron spectroscopy data records
SC4: Depth Profiling	ISO 14606: Otimization of sputter depth profiling TR 15969: Measurement of sputtered depth
SC5: AES	AWI 18118: Guide to use of experimental relative sensitivity factors for the quantitative analysis of homogeneous materials (AES & XPS)
SC7: XPS	ISO 15472: Calibration of energy scales (XPS) DIS 17973: Calibration of energy scales for elemental analysis (AES) DIS 17974: Calibration of energy scales for elemental and chemical state analysis (AES) CD 19318: Reporting of methods used for charge control and correction (XPS) WD 18327: Unintended degradation by X-ray photoelectron spectroscopy

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

qualitative analysis can be performed. COMPRO is available without charge and can be downloaded over the internet. Dr. Yoshihara was responsible for the design and development of COMPRO that has evolved in response to needs and to comments from many scientists in the VAMAS states (the present version is 6.52). This software is a major accomplishment and has considerable benefit. It has also enabled the development of a spectral database by the Surface Analysis Society of Japan that can also be downloaded without charge.

Prof. A. Jablonski (Institute of Physical Chemistry, Polish Academy of Sciences) has been appointed as an Observer to TWA 2. Prof. Jablonski has extensive experience in

quantitative AES and XPS as well as expertise in analyzing the effects of elastic-electron scattering in AES and XPS. He has already contributed to project 3, and I expect that he will also contribute to project 14(d) as well as provide useful advice on other projects.

The Chairman, Dr. C. Powell, participated in a combined meeting of the VAMAS International Steering Committee and Workshop for TWA Chairmen to be held April 9-11 in Gaithersburg, MD, USA. He presented a report on linkages between TWA 2 and standards development organizations. This report described some of the ISO standards that have resulted from TWA 2 projects as well as the incorporation of results from projects 14(c) and 14(d) in a new European project to develop improved capabilities for synchrotron microanalysis.

Technical Working Area 3

CERAMICS FOR STRUCTURAL APPLICATIONS

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In May of 2000 Mr. George Quinn stepped down as the chair for TWA 3, and the new chairperson, Dr. Kristin Breder of Saint-Gobain Abrasives, Worcester, USA, was approved by the Steering Committee. Mr. Quinn's hard work over many years is an important reason for the success of this working group.

The round robin on Elevated Temperature Flexural Strength testing was completed and the report issued by Japan Fine Ceramics Center (JFCC). Information about the findings in this round robin was reported to the ISO TC 206, Working Group 8 for inclusion in the draft standard on elevated temperature flexural strength testing. A new round robin on Determination of Phase Composition and Percent Crystallinity in Hydroxyapatite was approved by the Steering Committee, and questionnaires were sent out to potential participants regarding their capabilities. The report from the round robin Determining the Fracture Toughness of Ceramics Using the Single-Edge-V-Notched Beam (SEVNB) Method was published as a joint VAMAS/ESIS (European Structural Integrity Society) Procedure in June 2000. The method will be included in the new CEN Committee TC 184 standard test method for determination of fracture toughness.

Status of Current Projects

Project 13. High Temperature Flexure Strength.

The High Temperature Flexure Strength project was finished in August 2000, and the report was issued by Japan Fine Ceramic Center. [A. Okada and M. Mizuno, “VAMAS Round Robin on Flexural Strength of Silicon Nitride at High Temperature,” ISSN 1016-2186, Japan Fine Ceramic Center, Nagoya, Japan, September 2000.] Thirteen laboratories in six countries measured the strength of silicon nitride at 1200 ^{o}C in air. Semi- and fully-articulating fixtures were used. All testing was in four-point flexure, with either 10 mm x 30 mm or 20 mm x 40 mm spans. Most laboratories tested 10 or 12 specimens.

Among the numerous conclusions from this project were:

a. The limited number of specimens tested by each laboratory led to a large reproducibility uncertainty (between-laboratory strength variations). Much of the difference can be attributed to small sample size statistical effects; the differences are within the confidence intervals predicted by Weibull statistics.
b. Supplemental experiments confirmed that friction constraints may affect load-displacement curve data (and presumably the measured flexure strength) with fixtures having rollers that are not completely free to roll. Fixtures with rollers in square slots of insufficient clearance may inhibit roller motion.
c. Two laboratories performed additional testing in inert nitrogen environment. Nitrogen-tested specimens were weaker than air-tested specimens, presumably due to oxidative crack healing in the latter.
d. Load-displacement curves were valuable in interpreting the performance of the test fixtures and for confirming that the material has linearly-elastic behavior up to fracture.

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

Representatives from many companies, universities, government laboratories, and research institutes have expressed interest in participating in this round robin exercise. 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 hydroxyapatite (HA) crystallinity and phase composition. This new method differs from the usually cited method and potentially removes uncertainties stemming from overlapping peaks. HA is a ceramic material increasingly used as a biocompatible monolithic or coating encouraging adaptation of an implant into the human body, but the body's response depends critically on the phase composition of the material. Calibration specimens and specimens composed of mixtures of Ca-PO_4 glass, HA, CaO, α-TCP (tri-calcium phosphate) and β-TCP will be supplied for analysis. For parties interested in full participation (including producing a glass powder for preparing the calibration specimens) HA, CaO, α-TCP and β-TCP powders for preparing calibration specimens and specimens composed of mixtures of Ca-PO_4 glass, HA, CaO, α-TCP and β-TCP will be supplied for analysis.

The primary VAMAS output will be a report detailing the findings of the round robin, and giving an assessment of the reliability of the method proposed for the International Standard. The primary standardization output will be a statement of confidence in the technique written into the standard based on the findings of the round robin. Two International Standards for hydroxyapatite ceramics and hydroxyapatite coatings are currently at the committee draft stage of ISO/TC150/SC1. The purpose of these two documents is to specify the key characteristics that define hydroxyapatite materials and to stipulate the requirements that shall be met to allow their use as materials for surgical implants.

Proposed Future Projects

Projects from last years annual report that were in the very early stages or discussion phase have not materialized into formal projects. One new project is being initiated in the discussion phase:

Dr. S. J. Cho, of the Korea Research Institute of Standards and Science is interested in a possible round robin related to the strong influence of laboratory humidity and temperature on the flexure strength of certain ceramics, especially alumina. In many national standards, it is recommended only to record the laboratory temperature and humidity. In ISO FDIS 14704, it is recommended to flow nitrogen gas during testing or to coat specimens with oil before testing. A discussion note from Dr. Cho has been disseminated.

Technical Working Area 5

POLYMER COMPOSITES

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

Project 1, Assessment and Recommendations to ISO on Mode II Test Methods, has been completed. The project was organised to assist ISO TC61/SC13/WG16 in overcoming a veto on Mode I progression without Mode II. Based on the Mode II round robin it was recommended and agreed at the September 1999 ISO TC61 meeting that the "4-point ENF" procedure is the preferred method for standardisation. No further progress since ISO TC61 accepted preferred method for the Mode II standard. Work unable to progress, as difficulties exist due to AFNOR requirement on financial funding of individual test methods.

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

At this time, the first five steps in the program have been completed. Most of the top researchers in the field agreed to participate in the round robin (step 1). A recommended test procedure was developed with the help of these participants (step 2). A model system involving a specific fiber, resin, and preparation procedure was developed. Specimens were prepared and tested with the recommended procedure to be sure there were no problems (step 3). Samples for the round robin were prepared and distributed to participants. As of this date, 6 laboratories have completed the tests and returned their results (step 4). A number of characterisation experiments have been performed on the model system, both the resin and the fiber (step 5).

With the 6 laboratories that have responded to date, we feel the response is adequate although not optimum. Consequently, additional data will be sought. Meanwhile, the results already in hand will be analysed with the collaboration of the Statistics Group at NIST and final results should be available by the end of 2001.

Project 3, Measurement of Through-thickness Testing Properties, has been delayed. The development of composite materials for structural applications in new sectors such as the

ground transportation industry requires the use of thicker structures made of cost-effective materials (thick glass-epoxy woven composites, for instance). However, knowledge of through-thickness properties becomes necessary in order to design such structures and only few poorly understood methods are available. Therefore, there is an important need from industry to have such through-thickness test methods available.

Work has not started, as the EU Framework 5 network proposal to support the VAMAS project submitted in March 2000 was unsuccessful. NPL has a new project (starting 1/4/2001) on round-robin validation of the drafts previously prepared for through-thickness tension, compression and shear test methods, and therefore may be able to take on the leadership of this project.

Potential new topics include:

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

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

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

- Composite adhesion tests (peel-static/dynamic, weathering, edge delamination (fatigue), heat cycle) related to:

  a. composite bonded repair of structures, or
  b. composite stiffened conventional materials. (Proposed under IEA/OECD Climate Initiative (CTI) by Japan).

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

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

- Processing properties test methods.

COMPUTERIZED MATERIALS DATA

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This year was very successful for TWA-10. The VAMAS TWA-10 workshop "Generic Platform of Materials Database" was held and the Key-person's meeting confirmed the strategy and action plan of the international collaborative project on " Investigation on the generic platform for electrical data sharing system of materials data".

1. VAMAS TWA-10 Workshop

The VAMAS TWA-10 workshop on "Generic Platform of Materials Database" was held from February 2 to 5, 2001 at Osaka and Tsukuba in Japan. Thirty-five researchers from Japan, U.S.A. U.K. and EU participated and 18 papers were presented. MatML was introduced by NIST(USA) as the kernel of the generic platform of materials database. Implementation of MatML and XML to concrete materials database was presented by NMC (Japan). The possibility of the methodological application by STEP and the application for design with the Material Selection system were discussed. The program and the presented slides are put on the TWA-10 web site, (http://www.nims.go.jp:8080/vamas_twa10/)

2. Key-persons' Meeting

The key-persons' meeting was also held during the workshop. The meeting is summarized as follows:

1) MatML is recognized as the kernel of Generic Platform of materials database.
2) The first stage of implementation of MatML to the concrete materials database has been started.
3) The practical utilization of MatML with XML to databases will be collaboratively shared between: JRC (static strength), NIMS (creep strength), NMC (fatigue), NIST (ceramics [tentative]).
4) The importance of application software was recognized, and the description with STEP was expected to apply CAD/CAM.
5) The time schedule of the collaboration program was revised to correspond to these actions.

3. STEP Terminology Review

The application protocol on Aluminum Casting will be the subject of the first case review. Dr. John Carpenter (DOE) is preparing the application protocol. The topic of Life Cycle

Analysis is another possibility for an application protocol. Dr. Anna Moreno has established liaison between the CASCADE project and ISO TC 207.

4. Publications and Presentations

"Possibility and Problems of Generic Data-Sharing System of Materials' Database by Use of XML", K.Halada & H.Yoshizu, NRIM, 17th International CODATA Conference

"MatML: A Mark-up Language for the Exchange of Materials Data", E.F.Begley, C.P.Sturrock & J.Rumble, NIST, 17th International CODATA Conference

"Portability of Materials Data Evaluation Models using XML/Java", Y.Monma & H.Fujiwara, Kochi Univ. of Tech, 17th International CODATA Conference

The following papers were presented in the VAMAS TWA-10 Workshop, February 2001:

a) "Database and Designing" S. Iwata, RACE Univ.of Tokyo
b) "Progress in Materials Selection", C. Seymour, D. Cebon & M. Ashby, Granta Design and Cambridge Univ.
c) "Introduction of Database of Amorphous Metals in New Materials Center", A. Ueno, Toyota Tech.Inst.
d) "Introduction of Database of Heat Resisting Alloys by New Materials Center", K. Isonishi, Shiga Univ.
e) "Development Status of Materials Database at JRC Petten", H.H. Ove, IAM, JRC Petten
f) "Data Analysis System of Material Properties with XML and Java", T. Tsujikami & H. Horikawa, Ryukoku Univ.
g) "Material Cruise System: development of experiment system that should make open use of material database possible", Y. Oyatsu, T. Shuto & M. Tsuchiya, MRI
h) "Possibility of Computerized Materials Data Sharing System", K. Halada & H. Yoshizu, NRIM
i) "Progress of MatML", J. Rumble, E.F. Begrey & C.P. Sturrock, NIST
j) "STEP - the industrial context for materials data?", T.M. King, LSC Group
k) "Sharing Scientific Data, Program and Knowledge with XML", T. Ashino, Y. Ohtani & T. Saitoh, Knowledge System Council
l) "Activity for Materials Database Sharing", Y. Monma, Kochi Univ. of Tech.
m) "XML-based System Integration Method in Manufacturing Applications", T. Kojima & S. Ohtani, MEL
n) "Data-Free-Way and Data Exchange", J. Kinugawa & M. Fujita, NRIM
o) "Environmentally conscious materials selection - Electric Appliances -", N. Shibaike, Matsushita Research Inst.

Technical Working Area 13

LOW CYCLE FATIGUE

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The VAMAS/EC project, Quantifying Data Uncertainties and the Validation of a Code of Practice for the Measurement of Bending in Uniaxial Fatigue Test Pieces, has been completed. The objectives were: (a) to develop a framework for quantifying measurement uncertainties in low cycle fatigue data, (b) to validate the new Code of Practice for the measurement of bending caused by load misalignment in uniaxial fatigue testing, and (c) to provide recommendations for a best practice guide for routine low cycle fatigue testing of metallic materials. The final report for this project has been produced. A meeting of VAMAS TWA 13 national representatives was held in tandem with the EGF13 conference, held in San Sebastian, Spain, on 6-9 September 2000. It was agreed to review the VAMAS/EC draft report titled “A Guide for the Verification of Alignment of Uniaxial Test Systems” and to publish it as an ISO TTA document.

The above-mentioned report will also represent the basis of the international standard for fatigue test machine alignment. This was agreed during the last meeting of the ISO/TC164/SC5 Committee, which was held in Berlin in June 2000. The Committee approved a resolution that a new working group, ISO/TC164/SC5/WG11, is to be formed under the chairmanship of Dr F Kandil to produce the standard.

The chairman of TWA 13 is maintaining close cooperation with the ASTM E28.04.03 international forum that is currently revising ASTM E1012. The aim of this cooperation is to ensure that there will be no conflict between the VAMAS/ISO recommendations and the ASTM standard.

Another success to report is the adoption of VAMAS recommendations in the revised JSME standard. See publication below.

A New Publication

M Sakane and K Yamaguchi, “High Temperature Low Cycle Fatigue Standard Testing – JSME Recommendation”, Fourth Japan-China Bilateral Symposium on High Temperature Strength of Materials, 11-13 June 2001, National Research Institute for Metals (NRIM), Tsukuba, Japan.

METAL MATRIX COMPOSITES

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

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

1) SiCw / A2009 MMC

This material has been previously examined in the tensile testing exercise detailed in VAMAS reports 20 and 27. The round robin fatigue tests have been carried out at room temperature and elevated temperature (200 ^{o}C) at a test frequency of 10 Hz. The fatigue stress ratio selected was R = 0.1, and maximum stress levels of 500 MPa and 430MPa were chosen for the room temperature and elevated temperature tests respectively. To date 4 organisations have returned their test results, but one further set of data is expected. Each organisation carried out 3 tests under each condition according to their own test procedures.

The scatter in the data from room temperature tests was larger than that obtained at elevated temperatures. The data will not be analysed in detail until the outstanding set of results are received. In many cases the specimens failed outside the gauge length close to the grips, which indicates that the specimen geometry chosen may have contributed to the scatter in the results. Because of the form of the MMC (thin sheet) only flat test pieces could be manufactured, but this led to difficulties in polishing and many of the specimen failures appear to originate from the corners of the test piece. The data will be presented at the TWA 15 meeting but it is unlikely that a second exercise will be carried out.

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

The preliminary fatigue test data for small specimens (2.5mm in width) obtained from tests at NRIM were discussed with Prof. W. S. Johnson. It was agreed that larger specimens should for be used in the intercomparison exercise because the fracture mechanism for the small specimen could be considerably different from that for large size one. NPL has

subsequently machined specimens 10 mm wide from the sheet material and baseline fatigue tests have been carried out at 480 ^{o}C at Georgia Tech (USA) to establish the conditions for testing. The fatigue exercise will be started once the questionnaire returns have been received. It is hoped that between 6-10 organisations will participate.

3) SiC fibre /Al MMC

3M Material from the USA has been supplied by NRIM in the form of sheets approximately 150 x 70 x 1.5 mm. Nondestructive tests of all materials have been carried out using the ultrasonic C- scan technique by Dayton University (USA). At present no test programme has been agreed for this material. A new work package will be proposed as a result of the current survey and questionnaire.

4) SiCp / Al MMC

NPL has acquired a supply of commercially available material from Advanced Metal Composites (AMC) for inclusion in the activities of TWA 15. A new work package will be proposed as a result of the current survey and questionnaire.

Proposed Future Projects:

A number of areas for future work on MMC have been discussed at previous TWA 15 meetings in Italy and France, and suggestions for new work areas included fracture toughness, creep, compression testing, microstructural characterisation and thermal property measurements. To try and prioritise the test requirements, a new questionnaire has been prepared and distributed to the European members of the MMC ASSESS network. The same questionnaire will be circulated to all national representatives for comment and input.

Standardization Activities and Dissemination

Contact with ASTM continues with representation on the ASTM D30 committee on MMC, and NPL is also actively involved in a European Thematic Network on MMC - “MMC ASSESS”, co-ordinating the effort on test methods and mechanical properties. Two meetings have been held in the last 12 months - in Turin in October 2000 and in Cambridge in April 2001 - at which the activities of VAMAS TWA 15 were presented. A dedicated web site has been set up and Dr Lord has submitted details of the VAMAS TWA 15 activity and a link to the VAMAS web site for inclusion, which will provide further opportunities for the dissemination of the TWA 15 and VAMAS activities. The UK dissemination activities continue to be supported by DTI-funded projects at NPL and via the Institute of Materials UK Forum on MMC and Composites Division Board.

Technical Working Area 16

SUPERCONDUCTING MATERIALS

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

In Project No. 1, Bending Strain Effects on Critical Current in Oxide Superconductors, the objective is to establish a standard measurement method for the critical current in Bi-2212/-2223 oxide superconductors at 77 K with no external field, after bending each of the specimens to a different, designated curvature at room temperature. Round robin testing is underway among 12 participant laboratories. Three kinds of Bi-2223 superconducting tape samples and a set of bending tools were delivered to each of the participants in November 2000. Measurements should be done by the end of March 2001 and Results will be summarized by May 2001. After successful completion of the round robin, it is anticipated that a draft standard will be proposed to the IEC.

The objective of Project No 2, Measurement Method for Critical temperature in Oxide Superconductors, is to establish a standard measurement method. Round robin comparison testing was carried out using resistive, dc, and ac magnetization methods. Following analysis of the results, it was concluded that the resistive method should be recommended as the standard method. An IEC standard draft for this critical temperature measurement method was proposed as a committee draft at the IEC/TC90 meeting in March 2000. Modifications have been made on the scope, definition and measurement details. It is expected to move to the CDV stage at the next meeting in September.

In project No. 3, Measurement Methods for Trapped Field and Levitation Force in Bulk Oxide Superconductors, the objectives are to develop standard measurement methods of trapped flux density and levitation force. The trapped flux density profile of a bulk is measured by scanning a Hall probe on the surface of the specimen. The magnetization process in which a strong field is applied to a specimen during cool-down performs the flux trapping. For trapped-field measurements, the trapped flux density is strongly affected by the geometry of the specimens, which is known as the de-magnetizing effect in conventional permanent magnets. It has been clarified that such effect can be corrected by a simple calculation method. The correction curve was made for standardization.

Preliminary studies were made on trapped flux density and levitation force measurement methods. During trapped flux density measurements, a problem with specimen

breakdown occurred during the magnetization process. The problem was solved by impregnating samples with a resin that prevented the sample from cracking. An IEC standard draft for the measurement of trapped flux density was proposed at the IEC/TC90 committee meeting in March 2000 and the final working draft will be discussed at the next meeting in September 2001.

In Project No. 4, Measurement Methods for the Surface Resistance in Thin Film Superconductors, the objective is to validate an IEC draft standard, IEC 61788-7 on the surface resistance in thin film superconductors. The surface resistance is determined by applying a microwave signal to a cylindrical dielectric resonator sandwiched between two superconducting thin films and measuring the insertion attenuation of the resonator at a certain frequency between 8 GHz to 30 GHz. A round robin test program (RRT) was initiated with the goal of reaching an accuracy of 10 percent. The second RRT (domestic) was implemented in July 2000, in which a unique pair of sapphire rods was used to minimize the error from dielectric inhomogeneity. The resulting data scatter was less than 5 % and small enough. The third RRT is now in action and should be completed in 2001 by expanding the number of participants to 5. The IEC standard draft, IEC 61788-7 is now at the CDV stage. It will move to the FDIS stage according to the result of the voting which will be closed on May 25, 2001.

The objective of Project No. 5, Measurement Method for the Mechanical Properties of Oxide Superconductors is to establish standard test methods for mechanical properties such as yield strength and Young's modulus at room temperature. The measurement is performed at room temperature using a mechanical test machine. The first RRT was implemented in 2000 among 8 participant laboratories. In this RRT, the measurement conditions were not specified at all, but participants were requested to measure by their own techniques. For all three Bi-2223 superconducting tape samples, the data scatter (COV) of the 0.2% proof strength and the tensile strength was rather small among labs, while that of the Young modulus and elongation was large. The reasons for the large scatter are now under discussion. The next RRT will be implemented in a near future, where the measurement conditions are well specified.

A new project has been proposed to clarify the relation between V-I characteristics and flux pinning, to determine the relation among irreversibility (critical) fields obtained by different measurement methods, and to establish an adequate and reliable measurement method. Irreversibility field or critical field is one of the critical parameters, beyond which superconductors cannot carry any effective superconducting current. This parameter is important for practical applications as well as in material characterization.

Technical Working Area 17

CRYOGENIC STRUCTURAL MATERIALS

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A proposal of the draft for ISO/TC164/SC1 "Metallic Materials - Tensile Testing in Liquid Helium" has been approved as a New Work Item at the last SC1 meeting held in Berlin on June 20, 2000. The Working Document ISO/AWI 19819 was submitted to ISO TC 164/SC 1 in January 2001.

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

The objective of Project No. 2, Mechanical Tests in a High Magnetic Field, is to establish reliable methods of evaluating Young's modulus, yield strength, tensile strength, elongation, and fracture toughness at 4 K in a high magnetic field. Preliminary tests were carried out and the results indicated that the magnetic field had little effect on the load cell and/or extensometer. The specimens of titanium alloy for the first RRT were distributed to the participants and results have been reported from four participants so far. A TTA document for ISO/TC164 is in preparation.

Project No. 3, Advanced Fracture Toughness Test, is trying to develop an understanding and a J-integral testing method at liquid helium temperatures for a small-size round bar with a circumferential notch. Testing procedures and specimens for a round robin test program were distributed to participants. The results of the second round robin test on 9% Ni steel were analyzed and effects of the stiffness of testing machine were discussed for the validity of this testing method. A TTA document for ISO/TC164 is in preparation.

Technical Working Area 18

STATISTICAL TECHNIQUES FOR INTERLABORATORY STUDIES

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The role of TWA 18 is to support the work of the other TWAs by offering advice and statistical consulting. The primary responsibility for the statistical planning and analysis should in most case reside with the statisticians from the organization organizing or coordinating a project. This is necessary to secure the necessary close contact to the scientists responsible in the field of application. Members of the TWA or statisticians from the coordinating organization should be involved in new projects from the initiation of the project.

In general it is not intended for the TWA to initiate own projects. Objectives of the TWA are

- to provide statistical and computational support in the phases of the design and the 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), finite elements, and mathematical modeling 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. TWA 18 may give assistance finding a source for statistical consulting if it is not available within your institution.

Technical Working Area 20

MEASUREMENT OF RESIDUAL STRESS

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

Measurements and analysis of the results are now complete. During the past year a progress meeting was held at ISIS, Abingdon, UK from 13-14 July 2000 immediately after the Sixth International Conference on Residual Stress, which took place in Oxford. A draft protocol for making the measurements was discussed and has been circulated for final revision. Once the protocol has been agreed it will be submitted to ISO, ASTM and CEN for consideration as a draft standard. Each of the Standards bodies has identified appropriate committees to examine the draft. These committees are ISO/TC135, ASTM E28.13 and CEN/TC138. Initially, the protocol has been issued as an ISO/VAMAS/TTA document, ISO TTA 3, Determination of Residual Stresses by Neutron Diffraction. Following issuance of the ISO/VAMAS/TTA, it is envisaged that meetings will be held to introduce new potential users to the technique.

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

Publications for the period are as follows;

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

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

Technical Working Area 21

MECHANICAL MEASUREMENTS FOR HARDMETALS

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The completed TWA 21 project on bend testing led to a revision of an ISO standard, ISO 3327, Bend Strength Tests for Hardmetals. The revised standard has been adopted.

The current ongoing project is aimed at developing good practice in the toughness testing of hardmetals. Nine laboratories from 5 countries will participate in testing with corresponding support from a further 5 laboratories in 3 more countries. Most of the last year has been spent in the manufacture of test pieces, planning the test schedule and circulating batches of materials for test to participants. Ten batches of material from 4 organisations have been supplied for the evaluation of four test methods, including two geometries of the short rod/bar test, the Palmqvist test (where crack lengths at indentation corners are measured), and a notched beam test. A plan of the test outline is attached as Appendix A. The first results are expected by Autumn 2001.

Regular progress reports are given to

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

Future Project

A possible project has been identified based on the confusion between customers and suppliers in Europe and North America where specifications may require different hardness test methods. A comprehensive exercise to evaluate the underpinning reasons for the differences between Rockwell (depth sensing) and Vickers (indentation diameter) is likely to be well supported.

Appendix A

Hardmetal Toughness – TEST OUTLINE (Excluding Palmqvist tests)

Testing Organisation		TEST PIECE GEOMETRY			
		SB	SEB	SR1	SR2
NPL	SEB 2x5x35 mm		B(2) K(3) H(3)		
BAM	SEB 3x4x35 mm		B(2) K(3) H(3)		
Boart Longyear	SR2 10ϕx15 mm				H(3) B(2) K(3) P(1)
United Hardmetals	SEB 6x10x20 mm				
Teledyne	SB 12x12x18 mm	B(2) K(3) H(3)			
Kennametal	SEB 3x6x45 mm		B(2) K(3) H(3)		
Sandvik Hard Materials	SEB 2x5x35 mm		B(2) K(3) H(3)		
Marshalls	SR1 12.7ϕx19 mm			H(3) B(2) K(3) P(1)	
Hughes Christensen	SB 12x12x18 mm	H(3) B(2) K(3)			

Test piece design

SB - Short Bar, 12 mm x 12 mm x 18 mm
SEB - Single Edge Beam
SR1 - Short Rod, 12.7 mm ϕ x 19 mm
SR2 - Short Rod, 10 mm ϕ x 15 mm

Material Supply

B - Baildonit
P - Plansee TIZIT
K - Kennametal
H - Harditalia

Technical Working Area 22

MECHANICAL PROPERTY MEASUREMENTS OF THIN FILMS AND COATINGS

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Progress on analysis of data produced in the depth sensing indentation project (Project 1) has been slower than expected due to incomplete reporting by some partners. Some additional experimental work has now been carried out to provide more detailed information on some instruments with regard to the type of analysis software used. Analysis of the new data has now been completed and a draft report will be produced in April 2001.

The project on adhesion (Project 2) is continuing to be developed. Further experimental work has been carried out in a round robin conducted in Japan with TiN. Essentially, these results confirmed those previously obtained. The problem again was that the two coatings with “good” and “bad” adhesion behaved similarly. Further work is now being planned with different coating systems.

The objective of Project 1, Measurement of Hardness and Young’s Modulus of Thin Coatings Using Depth Sensing Indentation Instruments, is the development of methodologies for the determination of the properties of a coating *in situ*, and validation of the method by carrying out an international round-robin. Most of the data have now been collated in a form that allows comparison of the effects of the varying degrees of corrections implemented by some partners. When complete, this will give information about the sensitivity of the data to the different corrections, which involve thermal effects, instrument stiffness and indenter area functions. Additional analysis is in progress to examine the effect of using different algorithms to calculate hardness and modulus. There is some concern that, despite very careful work on the part of two partners using the same instrument, agreement is poorer than expected on one type of coating, so that the stability of the sample comes into question.

This work has raised a number of issues that cannot at present be resolved. A further round robin has been proposed by some participants. This would allow the experience gained in VAMAS and elsewhere to be used in defining a much more closely controlled intercomparison where agreement between laboratories can be better assessed. Information obtained in this project has been fed into standards now being written for depth sensing indentation in ISO and CEN.

The objective of Project 2, Adhesion of Thin Coatings, is to evaluate and compare several

test methods for adhesion of thin coatings (<10 μm), including bend testing and indentation, through round robin testing, with the long-term goal of developing a simple, quantitative engineering test for coating adhesion.

A survey was sent out on April 4, 2000, to approximately 200 people in order to gauge interest in the project and to help the organizer determine which coating systems and which adhesion tests are of greatest interest to the thin film community. To date, forty positive responses have been received, with suggestions for coating systems and tests to be studied. Preliminary results to the survey questions were discussed at the VAMAS 22 evening meeting on April 10 at ICMCTF00 conference in San Diego. Because of the very wide range of both coating-substrate systems and adhesion tests, the most likely course of action will be to perform several separate round-robins with several different coating systems.

An initial round-robin revealed that the first set of TiN coated samples were not suitable for the work, since it was not possible to differentiate supposed varying degrees of adhesion, by scratch, indentation or tensile testing. Further experimental work has been carried out with another set of TiN samples and the same result established. It is therefore concluded that either the tests cannot distinguish between different degrees of adhesion, which is considered to be unlikely, or that TiN coatings are not suitable for this work.

A second round robin, overseen by Takahito Ohmura in Japan, also looked at TiN coatings, on both steel and silicon wafers, using scratch and indentation techniques, plus tensile strain of the films on steel substrates. As with the initial round robin, scratch and indentation testing could not distinguish between “good” and “poor” adhesion in these systems, although results from tensile testing looked promising. The next step for the Japanese group will be to vary the adhesion of TiN on steel using heat treatment to grow thermal oxide on the steel substrate prior to TiN deposition. New work items on adhesion measurement have been agreed by CEN TC 184 WG5, and the progress to be made in this project will directly input into this activity.

No new projects have been formally proposed, but it is generally agreed that a project on residual stress measurement should be proposed, but only when Project 1 is complete. Dr. Uwe Beck at BAM has agreed to lead this work.

Technical Working Area 24

PERFORMANCE RELATED PROPERTIES FOR ELECTROCERAMICS

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

The concept of a new TWA in electroceramics pre-standardisation was taken in January 1999 and was approved at the Steering Committee meeting in 1999. The TWA is working to enlist the co-operation of measurement laboratories that are interested in evaluating methods for determining performance-related properties of electroceramics.

The remit of TWA 24 has been described to the IEEE standards subcommittee on Loss in acoustic materials (piezoelectrics) and has been accepted as a useful measurement good practice umbrella from which interlaboratory exercises may be managed. This provides a convenient path to the International Standards body, IEEE. Additionally, CENELEC standards subcommittee BTTF63-2, looking at developing standards in low and high power dielectric measurements of piezoelectrics, also have embraced the concept of VAMAS.

The first project that was to be led by Dr Takahashi of Japan Fine Ceramic Association – Piezoelectric coefficient via resonance method adapted from work of Hirose – has been delayed following an initial National Round Robin within Japan. The results of this work will be made available to the TWA Chairman, at which time a full International VAMAS style project may be properly formulated.

In light of the delays in the first VAMAS project a second project has been formulated. This project – now labeled ‘Project 1’ – has as title: *International Intercomparison of Direct Piezoelectric Coefficient using the Berlincourt Method.* This method is used frequently by

industrial users and producers of piezoelectric materials for quality control of ceramic pieces and materials data generation for design. A recent DTI funded, UK industry supported, piece of research carried out at NPL has identified a large amount of variability from one measurement to another, and from one organisation to another. This variability arises from the way the measurement is carried out, and NPL have developed ideas that describe the origins of this variability. It is entirely appropriate to extend this laboratory intercomparison to an international level through an initial Round Robin study. The method is used extensively worldwide, and the data generated would help us determine the real level of scatter in the determination of the direct piezoelectric coefficient via this method. Once the first round robin is finished then best practice will be assessed and a further round robin will be executed that demonstrates the effectiveness of our measurement method. A draft standard may then be written based on this technique and the results from our two Round Robin studies. It would be relevant to post this to relevant standards committees including CENELEC pre-standards committee BTTF63-2. The time plan is set out below:

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

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

Technical Working Area 25

CREEP/FATIGUE CRACK GROWTH IN COMPONENTS

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The aims of TWA 25 are to:

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

In the last year, two project meetings have been held. The first was in Japan in April 2000 with the Japanese VAMAS group in which the plans for extending VAMAS in Japan was discussed. The second general meeting was held in parallel with the HIDA 2 Conference in Stuttgart in October 2000. Over 15 institutional representatives attended the meeting. Nine oral presentations were made at the meeting. At the meeting it was noted that the Round Robin questionnaire had been collected from the experts in the field and that Imperial College will summarise and distribute the results. As part of a round robin exercise, test data was received on creep/fatigue crack growth for a Pipe and Plate geometry. The data was distributed among participants to analyze and evaluate the life of test pieces using their own methods. It was also suggested that in the next year specific papers will be produced by volunteers in the group and will be sent for special technical publication in a nominated Journal. The aims set out above were discussed and agreed upon. The details of the minutes, future meetings and additional information about participating information is contained on the VAMAS TWA 25 website at http://mesm.org

Previous/Current work

Title	Completion date	Output
Collect shared information on participants interest	Nov 99 to Nov 00	Data-base
Collect detailed information on creep and creep/fatigue component testing and analysis	On-going	Data-Base, Analysis and summary of experts comments
Planned Round Robin	On-going	Component life prediction

Other Planned Output in the Next 3 Years

- gather experts from industry and research institutes in order to identify their specific needs with respect to feature component testing.
- produce a data-base of available feature component tests and interpreted data.
- identify acceptable feature components and best practice for undertaking tests.
- produce recommendations for analysis of component data
- establish reliable methods for prediction of component behaviour.

Technical Working Area 26

FULL FIELD OPTICAL STRESS AND STRAIN MEASUREMENT

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This new technical working area was initiated in October 1999. Its objective is to develop standard equipment, algorithms, reference materials and procedures for full field optical stress and strain measurement. The techniques included are: photoelasticity, Moiré methods, laser speckle and interferometry methods, image correlation techniques, and thermal emission methods. The work will comprise collation of current best practice, the development of procedures where this does not exist and the production of codes of practice and guides to use. The plan stems from the need in industry to validate commercial and theoretical analysis, especially where complex structures and processes or novel methods are involved.

The first meeting of TWA officers and participants was held in June 2000 at the SEM conference in Orlando, Florida, USA. Presentations were given in the following areas:

1. Introductions (Eann Patterson , co-chair)
2. Rationale for Standards on Optical methods for Stress and Strain Measurement (Richard Burguete, co-chair)
3. Presentations:
   a. North American Perspective (Michael Sutton)
   b. European Perspective (Michel Honlet)
4. VAMAS TWA 26:
   a. Participants
   b. Modus Operandi
   c. Projects

This opportunity was taken to determine how to group the common areas of interest and

distribute projects accordingly. A web page with address www.twa26.org has been set up on the University of Sheffield server. The minutes of this meeting have been posted on the TWA 26 web page.

There are important current activities underway. First, an Expression of Interest (EOI) proposal was approved by the EU as part of the 5th Framework Programme. A dedicated call has been issued for proposals in the area of optical techniques for strain measurement. The consortium that will submit a proposal is being assembled. The call closes in February 2002. The dates for submission deadlines mean that new projects would start around June 2002. Second, a request for registration has been submitted to CEN/STAR for prioritisation of Co-normative and Pre-normative research.

Current project status

Projects have not yet been agreed but the 'areas of interest' have been updated as a result of the interests declared at the first TWA 26 meeting.

Proposed/Future projects

Discussion is proceeding to initiate a project which is based around the 'Standard guide for evaluating non-contacting optical strain measurement systems'. This guide is a draft standard submitted to the ASTM subcommittee 08.03 "Advanced apparatus and test methods" by Prof. Mike Sutton. A copy of this can be found on the TWA26 website under sutton.doc.

Technical Working Area 27

CHARACTERIZATION METHODS FOR CERAMIC POWDERS AND GREEN BODIES

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This Technical Working Area was approved in May 2000 and the kick-off meeting held November 9, 2000 at Sandia Labs, in Albuquerque, New Mexico. The objective of this TWA is to develop and standardize methods for the characterization of ceramic powders and green bodies because they are important for ceramic manufacturing processes. In this regard, the proposed work encompasses three different areas:

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

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

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

The results of this preliminary test have shown that the testing procedure has to be corrected with regard to a general applicability of the method to commercial powders and suspensions. However the results were promising and will contribute to a more reliable test method with a general applicability to suspensions rather than to powders. The results of the preliminary test have been discussed at a meeting of the participants, January 8, 2001 in Tsukuba, Japan. The measurement procedure is currently being refined. The project is related to ISO TC 206 NP 00.01

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

Technical Working Area 28

QUANTITATIVE MASS SPECTROMETRY OF SYNTHETIC POLYMERS

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With recent advances in mass spectrometry it is possible to measure the molecular mass of some biological and synthetic polymers. Among the advances is a variation of time-of-flight mass spectrometry, MALDI ToF MS, in which laser ablation is used to produce charged polymers in the vapor state. This technique has the potential to be an absolute method for measuring the molecular mass distribution of synthetic polymers, a long sought after goal in polymer characterization.

Industrial needs using the MALDI method varies. Quantitation of both a homopolymer and a mixture of the same homopolymer but with different end groups is of importance at lower molecular masses. This is the range of molecular masses for polymers, which are used as reactive prepolymers. The National Institute of Standards and Technology (NIST) in collaboration with American Society of Mass Spectrometry conducted an international interlaboratory comparison study of a polystyrene material by MALDI ToF MS. The Bundesanstalt für Materialforschung und –Prüfung (BAM) also conducted interlaboratory comparisons by MALDI ToF MS of polystyrene and other polymers for the purpose of evaluating classical methods. A large number of industrial laboratories participated in these interlaboratory comparisons. In the NIST interlaboratory comparison about half of the 23 laboratories reporting results were industrial laboratories.

As this is a new TWA we are focusing on soliciting laboratories to participate and concurrently finalizing measurements on a polymer sample used in the recent international round robin of characterization by mass spectrometry.

At present, two projects are under consideration:

a. Design a protocol for optimizing parameters for obtaining the best quantitative spectra from a MALDI TOF MS instrument for synthetic polymers.

b. Establish a protocol for sample preparation, data acquisition and analysis, building on results from the recently completed interlaboratory comparison.

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