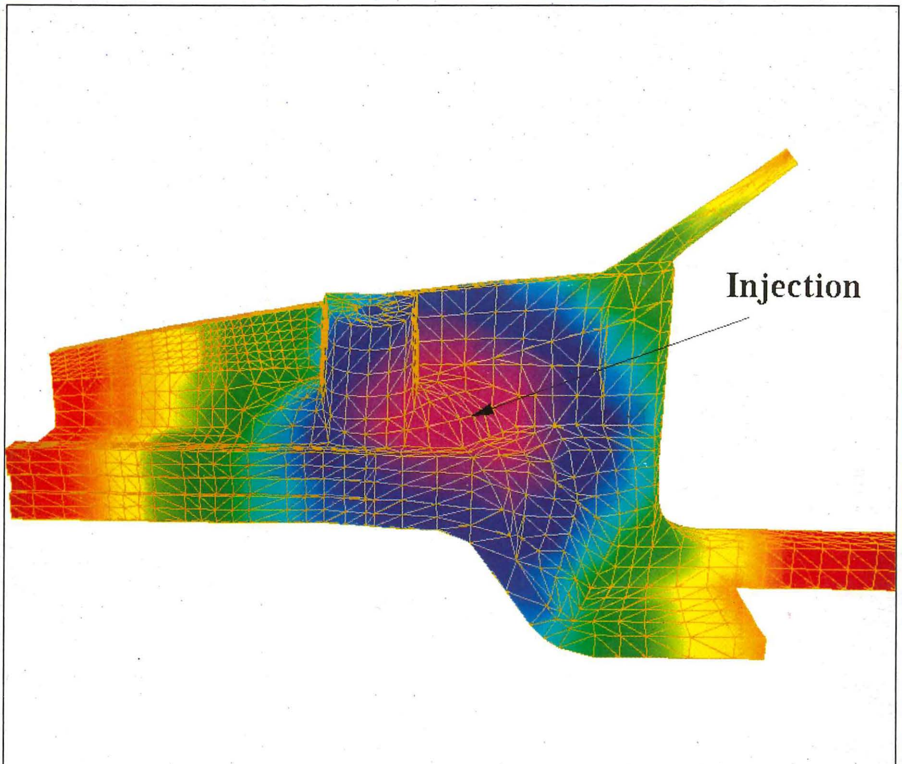
VAMAS



BULLETIN NO. 16
January 1993

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

VAMAS

The Versailles Project on Advanced Materials and Standards (VAMAS) supports trade in high technology products through international collaborative projects aimed at providing the technical basis for drafting codes of practice and specifications for advanced materials. The scope of the collaboration embraces all agreed aspects of enabling science and technology - databases, test methods, design methods, and materials technology - which are required as a precursor to the drafting of standards for advanced materials. VAMAS activity emphasizes collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardization action. Through this activity, VAMAS fosters the development of internationally acceptable standards for advanced materials by the various existing standards agencies.

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Cover: Finite element simulation of resin flow during liquid molding of a complex polymer composite component. The pink region shows earliest flow front, and red the latest.

Photograph courtesy of National Institute of Standards & Technology, Gaithersburg, MD, USA

VAMAS

• Table of Contents •

• Foreword •

At the conclusion of the June 1992 Steering Committee (SC) meeting in Berlin, the VAMAS chair and secretariat passed to the United States from the United Kingdom. As the new Chairman, it is my sincere pleasure to acknowledge the dedication and strong leadership provided to VAMAS by the past Chairman, Dr. Kamal Hossain. It is my intention and that of the new Secretary, Dr. James Early, to continue the high standards set by Dr. Hossain and Dr. Roebuck.

By way of introduction, I am the Deputy Director, Materials Science and Engineering Laboratory (MSEL), National Institute of Standards and Technology. I am a chemist by training, with extensive experience in analytical chemistry, analysis of materials and Standard Reference Materials here at NIST. The new Secretary is Scientific Advisor to the Director, MSEL, a metallurgist by training, with extensive experience in powder metallurgy, solidification, and fracture. We are both looking forward to working in support of the VAMAS mission and goals.

The SC has officially affirmed that the VAMAS organization has been re-authorized for five more years. The starting date of the extension is April 1992. The Memorandum of Understanding has been approved by six countries (Canada, Germany, Italy, Japan, UK and USA) and the Commission of the European Communities (CEC). A formal response from France has not yet been received.

Among the topics we plan to focus on during the next year are: (1) implementation of the new Thematic/Co-ordinator system for improving the administrative functions through delegation of certain authorities to reduce the delay for decisions between SC meetings; and (2) simplifying and strengthening the process for developing and obtaining substantive articles for the VAMAS Bulletin. These topics are described in greater detail in one of the feature articles in this Bulletin.

Beginning with this issue of the Bulletin, the section containing the Technical Working Area (TWA) progress reports will highlight those TWAs in which significant events or progress has occurred since the previous Bulletin issue. As experienced scientists and investigators, we know that research progress is uneven at best, with periods of time in which preparation is dominant and technical results premature. Progress reports which summarize only the status quo will no longer automatically be included in the Bulletin. We believe that this change plus the new sections, Standards Highlights and Recent VAMAS Outputs will sharpen the Bulletin focus and enhance information transfer. Standards Highlights will identify draft or adopted standards based all or in part on TWA work programs. The Recent VAMAS Outputs section will contain extended abstracts of VAMAS reports and articles.

Harry L. Rook
Chairman

• Standards Highlights •

In its role of providing the technical basis for drafting codes of practice and specifications for advanced materials, VAMAS fosters the development of internationally acceptable standards for advanced materials by the various existing national, regional, and international standards agencies. As VAMAS begins its second five-years, an increasing number of its pre-standards research projects have been concluded and the results transferred to standards-writing organizations. Although not every pre-standards research project was intended to produce definitive test results in direct support of a specific standards effort, we are now seeing the impact of VAMAS efforts through their recognition in an increasing number of adopted standards.

As part of a plan to provide greater visibility for the technical outputs of the Technical Working Areas (TWAs), two new sections have been added to the VAMAS Bulletin. This section, Standards Highlights, will identify draft or adopted standards documents from national, regional, or international standards bodies that are based all or in part on pre-standards technical outputs from VAMAS TWAs. In the absence of a central standards clearinghouse, VAMAS participants are strongly encouraged to notify the Secretariat of any such adopted standards and to send a copy to the VAMAS Secretary.

Recently adopted standards include:

1] ASTM E1457-92, ***Standard Test Method for Measurement of Creep Crack Growth Rates in Metals,*** approved February 15, 1992, ASTM Committee E24, Fracture Testing. VAMAS contributor - TWA 11, Creep Crack Growth, T. Gibbons, Chairman.

2] DIN 50 324, ***Prüfung von Reibung und Verschleiß Modellversuche bei Festkörpergleitreibung*** (Testing of friction and wear model test for sliding friction of solids), approved July 1992. VAMAS contributor - TWA 1, Wear Test Methods, S. Jahanmir and E. Santner, Co-Chairmen.

3] Draft Austrian Standard, ÖNORM M 8125, ***Prüfung von Reibung und Verschleiß Modellversuch: Kugel-Scheibe-Prüfsystem*** (Testing of friction and wear - model test: ball-on-disc system), approved November 1992. VAMAS Contributor - TWA 1, Wear Test Methods, S. Jahanmir and E. Santner, Co-Chairmen.

• Feature Article •

VAMAS: PHASE II (1992-1997)

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1 Background

An internal re-evaluation of VAMAS and its operating procedures has taken place concurrently with the effort to obtain formal re-approval of the VAMAS Memorandum of Understanding by the seven signatory countries and the Commission of the European Communities (CEC). The result of this introspective analysis by the Steering Committee (SC) has been a series of decisions designed to facilitate and enhance the work programs of the Technical Working Areas (TWAs) through a shift in specific decision-making authority away from the SC as a whole towards the TWA level.

2 Thematic Structure and Co-ordinator System

At the Montreal SC meeting in May 1991, a new mechanism for TWA coordination and oversight was proposed. The January 1992 issue of the VAMAS Bulletin (No. 15) summarized the initial recommended changes to the organizational structure and the approval process for new research projects, Technical Working Areas, and other related activities. At the Berlin SC meeting, additional modifications to the organizational structure and administrative procedures were made together with the formal implementation of the thematic structure for SC coordination with the TWAs. The basic process is based on the assignment to each TWA of one SC member, designated as the Co-ordinator, who has been delegated specific decision-making authority. The Co-ordinator acts on behalf of the SC for the TWA, based on the designated role and responsibilities, respectively, of the Co-ordinator and TWA Chair. Each SC member identified one or more TWAs for which they would accept responsibility as Co-ordinator. Assignments of SC members to most TWAs have been made and each TWA Chair has been notified of the initiation of the Thematic Structure/Co-ordinator system.

The SC Co-ordinator/TWA assignments as of April 19, 1993 are as follows:

Co-ordinator	Technical Working Area	
H. Czichos	TWA 1	Wear Test Methods
K. Hossain	TWA 2	Surface Chemical Analysis
J. Takagi	TWA 3	Ceramics
J. Martel	TWA 4	Polymer Blends
A. Iwahashi	TWA 6	Superconducting & Cryogenic Structural Materials
P. Giusti	TWA 7	Bioengineering Materials
G. Sievers	TWA 12	Efficient Test Procedures for Polymer Properties
D. Gould	TWA 13	Low Cycle Fatigue
K. Riley	TWA 14	The Technical Basis for a Unified Classification System for Advanced Ceramics
A. Oguchi	TWA 15	Metal Matrix Composites
[a]	TWA 5	Polymer Composites
[a]	TWA 10	Materials Databanks
to be assigned	TWA 8	Hot Salt Corrosion Resistance
not assigned	TWA 11[b]	Creep Crack Growth

Note: [a] Pending confirmation
[b] Terminated 9/92

The programmatic structure for the technical activities is organized around a series of umbrella themes. This five theme structure provides a coordinated framework for the description of the VAMAS technical programs. The TWAs have been grouped under the following themes:

I	METALS AND METAL MATRIX COMPOSITES	TWA 13, Low Cycle Fatigue TWA 15, Metal Matrix Composites
II	POLYMERS AND POLYMER MATRIX COMPOSITES	TWA 4, Polymer Blends TWA 5, Polymer Composites TWA 12, Efficient Test Procedures for Polymer Properties
III	CERAMICS AND CERAMIC MATRIX COMPOSITES	TWA 3, Ceramics

IV	TEST TECHNIQUES (non-material specific)	TWA 1, TWA 2, TWA 6, TWA 7, TWA 8,	Wear Test Methods Surface Chemical Analysis Superconducting & Cryogenic Structural Materials Bioengineering Materials Hot Salt Corrosion Resistance
V	MATERIALS CLASSIFICATION AND DATA	TWA 10, TWA 14,	Materials Databanks Unified Classification System for Advanced Ceramics

The management operation of the Theme/Co-ordinator system is based on the following:

- Each TWA will have a SC member assigned as Co-ordinator. The TWA Chair will directly report to the appropriate SC/Co-ordinator. The assignment of TWAs to a specific Co-ordinator is subject to periodic review to ensure appropriate technical match and distribution of coordination responsibility.

- The tracking and reporting of activities and progress in each TWA at each SC meeting, including receipt of Project Initiation Forms, would become the responsibility of the Co-ordinator or their designee.

- For proposed new projects, the Co-ordinator and TWA Chairman would review and approve the Project Initiation Form and then Fax the form to the other SC members with a two-week comment/rejection deadline. Minor corrections to the Form could be made by the Co-ordinator and TWA Chairman.

- The Co-ordinator, in concert with the TWA Chair, would initially establish realistic target completion dates for each project.

- The Co-ordinator would have the authority to approve participation by researchers/organizations from non-summit countries in TWA projects based on a written request describing the specific benefits and contributions expected from the participation. Written notification of action taken would be sent to the other SC members.

- The Co-ordinator would serve as a back-up source for written articles for the VAMAS Bulletin. The Secretariat would still depend on the TWA Chairs as the primary source of material for the Bulletin.

3 VAMAS Bulletin

One phase of our renewed effort to focus greater visibility on VAMAS activities is to highlight the reports, publications, and other outputs of the TWAs. In the past, each Bulletin issue contained work-in-progress reports from each TWA and one or more longer feature articles requested from specific TWAs. Under the revised administrative procedures described earlier, the TWA Chairs still retain primary responsibility for submitting status or work-in-progress reports on important developments in their various projects and substantive articles on completed projects for the VAMAS Bulletin. For future inclusion in the Bulletin, TWA Chairs will be responsible for submitting a one to three page summary or extended abstract for each formal TWA output, including final and interim VAMAS reports, scientific articles or articles on segments of completed projects that are to be published. This extended abstract or summary should be sent to the Secretary and would include: the purpose of the project; summary of results, conclusions, and recommendations; reference to where full report or article will be published; a brief bibliography of recent articles relevant to the VAMAS document; and, if appropriate, a discussion of the next step or phase. When finally published, two copies of the complete report or article should be sent to the Secretary.

4 Summary

The Chairman and I look forward to working with the active VAMAS membership for the next three years and appreciate the time and effort by the participants dedicated to making VAMAS a unique and effective pre-standards development body. The Steering Committee welcomes advice and suggestions on how to improve and strengthen VAMAS activities and increase the viability of VAMAS technical contributions to standards development.

• Feature Article •

ISO TECHNICAL COMMITTEE 201 ON SURFACE CHEMICAL ANALYSIS

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The International Organization for Standardization (ISO) recently created a new Technical Committee (ISO/TC 201) on Surface Chemical Analysis in response to a proposal from Japan. As of January 1993, ten national standards bodies had indicated willingness to become participating members of ISO/TC 201 and fifteen national standards bodies had indicated willingness to become observer members. The following subcommittees are planned: Terminology; General Procedures; Data Management and Treatment; Depth Profiling; Auger Electron Spectroscopy; Secondary Ion Mass Spectrometry; and X-Ray Photoelectron Spectroscopy. It is expected that working groups will be established for Glow Discharge Optical Spectroscopy and Total Reflection X-Ray Fluorescence Spectroscopy. ISO/TC 201 will develop international standards and will consider standards and documents prepared by other groups as potential international standards. This new ISO Technical Committee is a logical development that follows from the work of the VAMAS)Surface Chemical Analysis Technical Working Area 2 (SCA TWA2).

1 Introduction

In January 1991, the Japanese member body made a formal proposal to ISO to establish a new ISO Technical Committee (TC) on Surface Chemical Analysis. This proposal was balloted among the ISO member bodies and was approved by the ISO Council in November 1991 as ISO/TC 201. The secretariat for ISO/TC 201 was allocated to Japan. The first meeting of ISO/TC 201 on Surface Chemical Analysis took place in Tokyo on July 9-10 1992. This meeting was attended by delegates representing China, Germany, Japan, Poland, the United Kingdom, and the United States of America. This new ISO Technical Committee is a logical development that follows from the work of the VAMAS Surface Chemical Analysis Technical Working Area 2.

An independent proposal to establish a TC on Microbeam Analysis was submitted to ISO by the Chinese member body. This proposal was also approved, and the new TC was designated ISO/TC 202. The secretariat for ISO/TC 202 was allocated to the Chinese member body from whom further information on ISO/TC 202 activities can be obtained.[1]

2 Purpose and Structure of ISO/TC 201

According to ISO procedures, all national member bodies (i.e. standards organizations) have the right to participate in the work of technical committees and subcommittees. There are two types of membership. The first type of membership (P-members) refers to members who intend to participate actively in the work; they have an obligation to vote on draft international standards and, whenever possible, to participate in meetings. The second type of membership (O-members) refers to members who intend to follow the work as observers; they receive committee documents and have the right to submit comments and to attend meetings.

Table 1 lists the nations whose standards bodies have elected to become members of ISO/TC 201 (as of January 1993) and also shows the name of the national standards body for each country. There are currently 10 P-members and 15 O-members. Scientists of the 25 countries listed in Table 1 can learn of ISO/TC 201 activities and can seek to participate in the deliberations leading to international standards by contacting their own national standards body. Scientists of other countries should contact their national standards body if they believe it advantageous for their country to be represented on ISO/TC 201. The names and addresses of other national standards bodies can be obtained from the authors or from the ISO/TC 201 secretariat.[2]

Delegates at the Tokyo meeting of ISO/TC 201 approved a Strategic Policy Statement for the new Technical Committee. This document identifies Auger Electron Spectroscopy (AES), Secondary Ion Mass Spectrometry (SIMS), and X-Ray Photoelectron Spectroscopy (XPS) as the three techniques most commonly used for practical surface analyses. Four other techniques (Glow Discharge Optical Spectroscopy [GDOS], Sputtered Neutral Mass Spectrometry [SNMS], Fast Atom Bombardment Mass Spectrometry [FABMS], and Total Reflection X-Ray Fluorescence Spectroscopy [TXRF]) were identified as being suitable for certain applications. Finally, sputter depth profiling was identified as a common application of surface analysis techniques for determining composition versus depth.

The Strategic Policy Statement describes anticipated future trends in the field of surface chemical analysis. Continuing demands are made for surface analyses with higher spatial resolutions (both parallel and normal to the surface). Although most present surface analyses are now qualitative, there are increasing needs for quantitative analyses. It is also then necessary to identify the surface chemical phases present in the specimen volume being analyzed and to determine their degree of homogeneity. Commercial instruments are now being supplied with increasingly powerful computer systems and software. There are needs to validate and extend algorithms used for the processing of measured data. Databases are becoming available, but their quality and reliability need to be assessed on a continuing basis. It is expected that recommended procedures, reference data, and reference materials will be used to an increasing extent to ensure that surface analyses can be made with the desired precision, accuracy and efficiency. Standards are needed to ensure the reliability of the techniques in common use.

The ISO Technical Board has approved the following official scope for ISO/TC 201: "Standardization in the field of surface chemical analysis, which uses electrons, ions, neutrons or photons both as an incident beam and a detecting signal". With current techniques of surface chemical analysis, compositional information is obtained for regions close to a surface (generally within 20 nm) and composition/depth information is obtained with surface analytical techniques as surface layers are removed.

It is proposed to establish the subcommittees listed in Table 2 and it is expected that working groups will be established for Glow Discharge Optical Spectroscopy (GDOS) and for Total Reflection X-ray Fluorescence Spectroscopy (TXRF). Further details on the planned technical work for the subcommittees are given below, but here we point out that the proposed General Procedures Subcommittee is expected to engage in standardization of the procedures common to two or more subcommittees of ISO/TC 201 such as specimen preparation and handling, specification and preparation of reference materials, and methods of reporting results. The Data Management and Treatment Subcommittee is expected to be active in the standardization of data bases, for the transfer of data between instruments, and for specifying the properties of algorithms used for surface chemical analysis.

3 Proposed Activities of ISO/TC 201

It is expected that ISO/TC 201 will consider existing standards prepared by national standards bodies as potential international standards, will establish liaisons with relevant international groups, will consider documents prepared by such international groups as new work items for potential international standards, and will develop new international standards. Whenever possible, recommendations from these organizations will be considered by ISO/TC 201 as the basis for potential draft international standards.

At present, the ASTM Committee E-42 on Surface Analysis is the only national standards body that has developed standards for surface analysis.[3] According to ISO procedures, ISO/TC 201 has invited the U.S. member body to propose formally that existing ASTM E-42 standards be considered for the so-called `fast-track' approval procedures for ISO standards. The fast-track procedure is a means for developing international standards on an appreciably shorter time scale compared to the normal procedure.

Two international groups are currently developing recommendations for surface analysis, the Surface Chemical Analysis Technical Working Area (SCA TWA2)[4] of VAMAS and the Working Group on Surface Analysis (WGSA)[5] of the International Union of Pure and Applied Chemistry (IUPAC). Formal liaisons are proposed between ISO/TC

201 and each of these two international groups. ISO/TC 201 has invited VAMAS to propose formally that documents prepared by the VAMAS SCA TWA be submitted as new work items for consideration as ISO standards. A similar invitation has been made to IUPAC concerning documents prepared by the IUPAC WGSA. An important item in the ISO/TC 201 Strategic Policy Statement is that topics for standards to be developed by ISO/TC 201 will be chosen to avoid duplication of effort with the current work of existing organizations (such as the ASTM E-42 Committee, VAMAS SCA TWA2 and IUPAC WGSA).

Table 3 lists areas of work proposed for consideration by specific ISO/TC 201 subcommittees and working groups. It is clear from Table 3 that many more standards are needed than are available from the ASTM E-42 Committee and from the work of VAMAS SCA TWA2 and IUPAC WGSA.

4 Summary

The new Technical Committee ISO/TC 201 on Surface Chemical Analysis met in 1992 and has approved a plan for the development of international standards in this field. It is expected that subcommittees will be formed shortly, and that decisions will be made as to their first meetings (probably in 1993). Subcommittees will begin technical work in their respective areas, usually through the appointment of working groups on specific topics.

The formation of ISO/TC 201 is an indication of the success of VAMAS SCA TWA2 in identifying important needs for pre-standards research in surface chemical analysis and in developing a large number of international projects to address those needs. ISO/TC 201 will be the focus for standards development for surface chemical analysis on a world-wide basis and will naturally involve scientists from both VAMAS and non-VAMAS states.

5 Acknowledgement

The authors are indebted to Dr. J. Early, Secretary, VAMAS, for his assistance in the preparation of this article.

6 References

1. Prof. Lin Zhuoran, P.O. Box 2724, Beijing 100080, China (Fax: +86 1 256 4613).
2. Dr. T. Edamura, Japanese Standards Association, 4-1-24 Akasaka, Minato-ku, Tokyo 107, Japan (Fax: +81 3 3582 2390).
3. C. J. Powell, Surf. Interface Anal. **19**, 237 (1992).
4. M. P. Seah, Surf. Interface Anal. **19**, 247 (1992).
5. W. H. Gries, Fres. Z. Anal. Chem. **333**, 596 (1989).

Table 1. Listing of national standards bodies which are members of ISO/TC 201 on Surface Chemical Analysis (as of January 1993).

(a) P-Members

Austria	Österreichisches Normunginstitut
China	China State Bureau of Technical Supervision
Germany	DIN Deutsches Institut für Normung
Italy	Ente Nazionale Italiano di Unificazione
Japan	Japanese Industrial Standards Committee
Russian Federation	State Committee for Standardization and Metrology
Sweden	SIS - Standardiseringskommissionen i Sverige
Turkey	Turk Standardlari Enstitusu
United Kingdom	British Standards Institution
USA	American National Standards Institute

(b) O-Members

Australia	Standards Australia
Belgium	Institut Belge de Normalisation
Egypt	Egyptian Organization for Standardization and Quality Control
Finland	Suomen Standardisoimisliitto SFS
France	Association Française de Normalisation
India	Bureau of Indian Standards
Ireland	National Standards Authority of Ireland
Korea, Republic of	Bureau of Standards
Norway	Norges Standardiseringsforbund
Philippines	Bureau of Product Standards
Poland	Polish Committee for Standardization
Romania	Institut Roumain de Normalisation
Singapore	Singapore Institute of Standards and Industrial Research
South Africa	South Africa Bureau of Standards
Switzerland	Swiss Association for Standardization

Table 2. Proposed subcommittees of ISO/TC 201 on Surface Chemical Analysis.

1.	Terminology
2.	General Procedures
3.	Data Management and Treatment
4.	Depth Profiling
5.	Auger Electron Spectroscopy (AES)
6.	Secondary Ion Mass Spectrometry (SIMS) (includes Sputtered Neutral Mass Spectrometry and Fast Atom Bombardment Mass Spectrometry)
7.	X-Ray Photoelectron Spectroscopy (XPS)

Table 3. Proposed areas of work to be considered by the subcommittees (shown in parentheses) of ISO/TC 201 on Surface Chemical Analysis.

A.	Instrument Specifications (AES, GDOS, SIMS, TXRF and XPS)
B.	Instrument Operations (AES, GDOS, SIMS, TXRF, XPS, Depth Profiling, and General Procedures)
C.	Specimen Preparation (General Procedures)
D.	Data Acquisition (AES, GDOS, SIMS, TXRF, XPS and Depth Profiling)
E.	Data Processing for Qualitative Analysis (AES, GDOS, SIMS, TXRF, XPS, and Data Management and Treatment)
F.	Data Processing for Quantitative Analysis (AES, GDOS, SIMS, TXRF, XPS, and Data Management and Treatment)
G.	Reporting Results (AES, GDOS, SIMS, TXRF, XPS, Depth Profiling, General Procedures, and Data Management and Treatment)
H.	Terminology (Terminology)
I.	Reference Materials (General Procedures)

• Feature Article •

NEW TECHNICAL WORKING AREA TWA15 METAL MATRIX COMPOSITES

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1 Introduction

The term composite usually means a combination of two or more constituent elements to form a bonded quasi-homogeneous structure that produces synergistic mechanical and physical property advantages over that of the base elements. In today's technical society, the term composite almost always brings to mind a polymer or resin matrix composite. However, metal matrix composites are starting to attract more attention. Historically, metal matrix composites (MMCs) were among the first continuous fiber-reinforced composites studied. Systems such as steel wire reinforced copper were early model systems. Metal matrix composites available today usually conform to one of the following three groups: (1) a particulate-based system formed by the addition of small granular fillers to a binder that generally results in an increase in stiffness, and perhaps, some increase in strength; (2) a whisker/flake system that realizes a greater proportion of the filler strength due to the higher aspect ratio (length/diameter) of the filler and, hence, has a greater ability to transfer load; and (3) a continuous fiber system that, due to fiber continuity, realizes the full properties (strength and stiffness) of the high performance fiber. These three composite systems are shown schematically in Figure 1.

Each type of composite system has advantages and disadvantages. The particulate-based system offers the lowest cost, significant stiffness improvements, and nearly isotropic properties. However, the strength improvements are slight and the strain to failure and fracture toughness are on the low side. The whisker-based composites are more costly but do offer more strength, in general, than the particulate-based composites. The continuous fiber reinforced metal matrix composites offer the best combination of strength and stiffness; however, the cost of this system is very high.

2 Background

Prior to the late 1980s the interest in metal matrix composites had fluctuated between mild interest and no interest at all. The only production continuous fiber-reinforced metal matrix composite components of that era currently in service are the boron/aluminum tubular struts on the United States Space Shuttles. The main disadvantage of continuous fiber-reinforced metal matrix composites is the high cost of the fibers and of fabrication. Cutting and drilling of some of the current systems can be very expensive compared to traditional metal shop operations. Compared to resin matrix composites, MMCs may offer many attractive properties such as better environmental tolerance to moisture and temperature, higher interlaminar strength, and better impact and lightning damage resistance. Compared to normal homogeneous structural metals, MMCs offer much higher stiffness-to-weight and strength-to-weight ratios. However, in neither case could the advantages of MMCs justify the additional costs.

In the early 1980s, several areas of technological advancement sparked a renewed interest in continuous fiber-reinforced metal matrix composites, namely, the need for high temperature materials for aerospace structures, advanced engines, and the need for materials with a high degree of thermal dimensional stability for space antenna application. MMCs have unique properties that make these technology advancements conceivable. MMCs will still be very expensive, but the applications are such that polymer matrix composites and homogeneous metals may not, based on the present state-of-the-art technology, be able to meet the new requirements. MMCs are currently being used or considered for use in a number of applications ranging from structural materials on hypersonic aerospace vehicles to internal automobile engine parts. Continuous fiber reinforced titanium matrix composites (TMCs), for example, have the potential for very high temperature applications of 650 °C and above. These materials are quite expensive and are targeted for high performance aerospace applications. Discontinuous reinforced aluminum (DRA) matrix composites have much lower temperature capability, as well as strength and stiffness properties, when compared to the TMCs; however they can have a significant improvement in many mechanical properties over their homogeneous aluminum counterparts and their cost is not prohibitive. Therefore there may be many commercial applications for the DRA.

Since there are many new MMC systems under consideration and development to meet projected needs, the future looks promising. A few examples of future applications of MMCs are discussed below.

Space: Graphite-reinforced metal matrix composites are being considered for the next generation of high stiffness, low-thermal-expansion materials for applications on dimensionally critical spacecraft structures. The closer the coefficient of thermal expansion is to zero the better. High specific stiffness is also desirable. One such structure is the wrap rib concept space antenna. These materials offer many advantages over resin matrix composites, such as higher electrical and thermal conductivity, better radiation resistance and no outgassing. However studies to date indicate that weight and cost may be a problem.

Missiles: As a family of structural materials, metal matrix composites have great potential for missile airframe applications. Their high specific stiffness justifies their use on missile airframes. This increased stiffness is the increased Young's modulus coupled with a slightly lower density than found in comparable unreinforced metal alloys. The increased specific stiffness, and sometimes increased specific strength, results in airframe weight savings which mean improved performance. Weight savings are direct payoffs to missile systems in that they can be used for either increased velocity or for increased range. Increased range also results in increased launch aircraft standoff distances, enhancing the survivability of the launch aircraft. Reduced structural weight can also allow increased warhead payload which improves the lethality of the missile system. Lighter weight airframe can also permit the use of additional electronic "black boxes" which can improve the missile systems guidance, control, survivability, and overall mission effectiveness.

Since a missile is a non-man rated aircraft, with minimum fatigue design requirements, metal matrix composites can be used with minimum risk relative to issues such as fracture toughness. Therefore, those particulate and whisker-based MMCs that have rather low fracture toughness can be used effectively in missile airframe designs.

Automobile engines: Both discontinuous and continuous fiber reinforced aluminum matrix composites have been successfully used in recent years for making various internal parts for automobile engines. These composite materials have demonstrated excellent wear resistance. Since they are considerably lighter than conventional steel parts, lower dynamic stresses are generated in the moving parts. Cast DRA matrix composites are also being considered for engine blocks to reduce the overall weight of the automobile which should result in improved mileage and less pollution.

Hypersonic vehicles: Turning the dream of hypersonic flight (airspeed between Mach 6 and Mach 26) into a reality may be possible because of recent breakthroughs in both engine and materials technology and in the use of super computers. However, further development is required to create the light-weight, high strength material needed so that an Aerospace Plane will survive the extreme temperatures and pressures it will encounter.

The temperatures that will be encountered by hypersonic vehicles are much too high for polymer matrix composites. Indeed, the temperatures are too high for all but a few homogeneous metals. Of these few homogeneous metals, practically none have sufficient stiffness and strength at the higher temperatures to allow the structure to survive. However, by reinforcing some of these light, high temperature alloys (i.e. titanium and titanium-aluminides) with high strength, stiff ceramic or graphite fibers, the resulting MMC will have the capability to survive the harsh environments of aerospace travel repeatedly.

3 Issues/Problems

Metal matrix composites are unique because they combine the high stiffness and strength of fibers but also retain the elastic-plastic behavior of the matrix material. In high temperature applications, the metal matrix may exhibit viscoelastic behavior as well as develop significant thermal residual stresses as they are cooled down. MMCs also display unique fatigue and fracture behavior. Experimental testing procedures for MMCs are not currently very well developed or documented. Analytical material models are not currently available to handle all the particular problems (elastic-plastic, viscoplasticity, thermal stresses, etc.) that need to be addressed in designing with MMCs. Thus, much work remains in understanding and designing with MMCs.

4 Standards Activities

ASTM is currently working on the continuous fiber MMC testing methodology in ASTM Subcommittee D30.07 on Metal Matrix Composites. Round robin activities are currently under way in static tensile testing and fatigue at room and elevated temperatures. DRA composites are being addressed in ASTM Subcommittee E08.09 on Fracture of Advanced Materials and Composites which is currently conducting a fracture toughness measurement round robin. In addition to these ASTM MMC round robin activities, the UK (led by NPL) has conducted an interlaboratory round robin on tensile testing of DRA composites. The U.S. Air Force sponsored a round robin for testing DRA composites that are part of the Air Force's Title III DRA Program. The VAMAS TWA15 on Metal Matrix Composites has members involved with each of these cited efforts and will use this past experience and on-going efforts to complement any VAMAS activities in this area.

The first meeting of TWA15 was held March 31, 1992, at Churchill College, Cambridge, UK, and hosted by Dr. Neil McCartney, NPL. In attendance were representatives from France, Japan, Spain, UK and the USA. It was reported that Germany also intends to participate. A preliminary survey of potential VAMAS participants conducted in 1992 prior to the meeting indicated significant interest in fatigue testing; modulus measurement and data analysis; fracture toughness testing; and thermal properties measurements. After reviewing the survey results, the attendees proposed that the initial focus of the project address tensile testing of DRA composites at room and elevated temperatures because of the widespread interest in the topic and the availability of DRA materials. It was agreed that the TWA would review the results of the ongoing ASTM round robin activities on fatigue, fracture, and tensile testing on whisker and continuous fiber reinforced MMCs before considering the room and elevated temperature fatigue testing of continuous fiber reinforced metals (CFRM). Immediate efforts are exploring sources of CFRM for testing.

A second meeting of TWA15 was held November 17, 1992, in Miami, Florida, USA, in conjunction with the ASTM Subcommittee D30.07 on Metal Matrix Composites meeting. Representatives from France, Germany, Japan, UK and the USA attended the meeting. The progress of the ASTM activities and their coordination with the VAMAS activities, including specimen design and machining, and the number of tests per laboratory for the planned DRA round robin activity were discussed. Action items were given to various VAMAS attendees. The next meeting of TWA15 is planned for July 14, 1993, in Madrid, Spain, in conjunction with the ICCM meeting. For more information on TWA15, Metal Matrix Composites, and its developing program, interested parties should contact Dr. W. Steven Johnson.

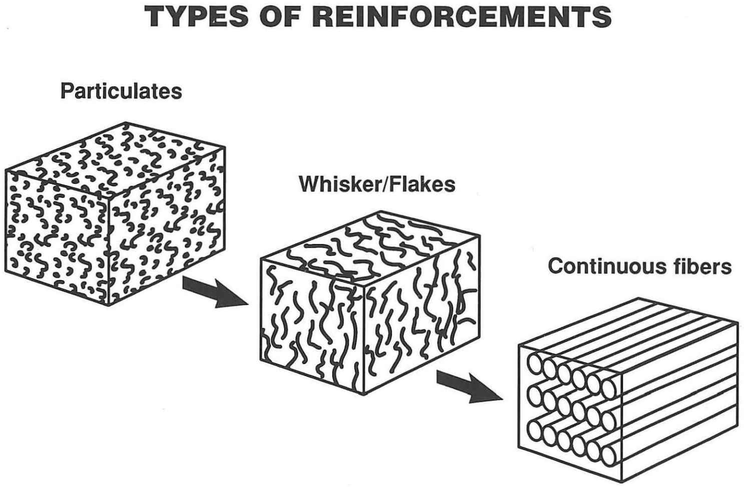

Figure 1. Schematic of Types of Composite Reinforcements

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 12, Final Report
"CEN-VAMAS Round Robin on Grain Size Measurement
for Advanced Technical Ceramics"
by L. Dortmans and G. De With, Centre for Technical Ceramics, Eindhoven,
the Netherlands; and R. Morrell, National Physical Laboratory,
Teddington, United Kingdom

The results of a joint CEN/VAMAS Round Robin on grain size measurement for advanced technical ceramics were analyzed. The round robin was established primarily to verify the equivalence of line and circle methods for manual determination of mean linear intercept length in a proposed CEN Euronorm, but also examined the possibilities of standardizing measurements of porosity and grain size distribution using overlaid grid intersection counting methods. Two fine-grained alumina ceramics with very different microstructures and a computer-drawn `Ideal' microstructure were used. Twenty-five participants from Europe, Japan, and the USA took part. VAMAS participation was coordinated by TWA3, Ceramics.

The results show that scatter levels of typically 20% between participants are obtained for the measurement of mean linear intercept length and the grain size distribution, and are primarily due to quality of specimen preparation, to selection of area for analysis, to interpretation of features observed and to a lesser extent, due to random positioning of lines, circles and grids on the micrograph. These levels of scatter are considered to be broadly acceptable, bearing in mind the inherent randomness of microstructures.

The results are believed to validate the equivalence of the line and circle methods for mean linear intercept size described in the draft CEN standard. The 30% scatter in the grid method porosity data is mainly due to the low level of very fine porosity in the nominally dense ceramic used and also to the variable interpretation of a complicated micrograph, and is explainable by simple random counting variations. The grid methods for grain size distribution measurement seem suitable in principle for implementation in future standards. However, in all cases, the quality of micrograph preparation and interpretation needs to be given further attention because these factors appear to be of major importance in achieving consistent results between laboratories. The results of this project have been used to prepare the new Euronorm: ENV 623 Methods of Test for Advanced Monolithic Technical Ceramics: General and Textural Properties, Part 3, Determination of Grain Size.

Three laboratories also applied image analysis systems. The results did not compare well with the line or circle data.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

BAM/VAMAS Technical Report No. 193/No. 13
"Statistical Consulting in the Framework of VAMAS: The Role of the Technical Working Area/Advisory Group 'Statistical Techniques for Interlaboratory Studies and Related Projects'",
by W. Gerisch, Th. Fritz and S. Steinborn
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1 Background

In connection with experimental R&D activities in science and technology, there is nowadays a great demand for statistical information. This refers, on the one hand, to sciences like agriculture, biology, chemistry, medicine, psychology, sociology, education and many other fields. On the other hand, driven by the need to compete on price and quality in the world market, an increasing demand for statistical advice rises from industry and government, where the enhancement of the effectiveness of engineering and industrial experimentation, in particular design, measurement, testing, specification and standards, came into the focus of statistical work.

These circumstances led to the development of new statistical areas like biometrics, chemometrics, quality control and engineering, reliability and operations research. Moreover, there was a strong and continuing impact on academic teaching of statistics towards the training of statistical consultants and, in particular, industrial and government statisticians, who differ, in general, from the pure statistician in knowledge, skill and attitude.

To be more precise, important attributes of an effective statistical consultant are the following: familiarity with statistical theory, practice and literature; qualification for extending and adapting statistical methodology to novel environment; familiarity with the client's subject matter, up to the ability of serving in the role of helping to identify and formulate his or her problem in quantifiable terms and observing the role of statistical tools; abilities of problem solving and effective use of computers; ability to adapt quickly to new problems and challenges; good communication skills; and ability to work in a timely fashion within real world constraints.

2 History

The response of VAMAS to this trend led to an investigation of the importance of statistical consulting from the viewpoint of the VAMAS collaborative projects, in particular its interlaboratory studies. During its Tokyo meeting (1991), the Steering Committee put onto the list of new TWA proposals a request to organize a group of statisticians, engineers, physicists and chemists with skills listed earlier and interests in materials research and engineering or related fields, which can realize the concept of statistical consulting in the frame of VAMAS. The decision was confirmed during the Berlin meeting (1992) of this Committee, and a corresponding group `Statistical Techniques for Interlaboratory Studies and Related Projects' consisting of 18 experienced consultants from G7 and non-G7 countries is now ready for confirmation by the Steering Committee.

3 Purpose

The main purpose of this report is to give the reader an impression of the importance of statistical consulting in materials research and engineering from the VAMAS viewpoint. In particular, it is intended to make the experimental investigator in the TWAs familiar with activities, services and tools to be offered by the new group. For the work of the members of the group, the report defines and discusses objectives, according to the following terms: service, optimization, research and consulting. These considerations, which amount to a working and research program, cover the essential aspects of statistical consulting in the frame of VAMAS. They focus the strengths and weaknesses of statistical models, approaches, guidelines and standards available for the support of interlaboratory research and testing, and include more than 260 references on more recent books, reports and papers. Finally, the report is intended as a document which describes not only scientific and technical aspects in connection with the role of the new group in the frame of VAMAS, but also gives details of the organizational work within the group up to the decisions and results of its initial meeting in Berlin (27th November 1992).

4 Summary

In the report, Chapter 1 contains references for further studies on advanced materials for the intended statistical consultant. A provisional classification of interlaboratory studies based on Horwitz's classification (1988) shows that additional work on classification, terminology and standardization is necessary in the field of interlaboratory research. Finally, the objectives of the group are laid down: statistical service for the TWAs, in particular in the field of interlaboratory studies; optimization of specimens and test devices by classical and more recent methods (Taguchi); statistical research; consulting in mathematics and computational statistics. Chapter 2 starts with a detailed discussion of these objectives and contains, in particular, a list of 18 problems or fields for corresponding statistical pre-standards research. Organizational aspects of the group are the focus of Chapter 3. Chapter 4 reviews the extensive background of collaborative activities in science and technology. The minutes of the initial meeting of the group are reprinted in Appendix A. In particular, a detailed list of members, reports on institutes and work, a list of possible statistical contributions and tools, a discussion of the program and a resolution to VAMAS are presented. Appendix B summarizes the contents of the report and also mentions plans to organize conferences in the more general new field of computational materials research and engineering.

• Technical Working Areas •

Technical Working Area 1

WEAR TEST METHODS

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Objectives

- To develop internationally agreed wear test methodologies for advanced materials, initially ceramics and inorganic coatings
- To improve reproducibility and comparability of wear test methods
- To characterize wear behavior of advanced materials

1 Background

In support of the mission of VAMAS, the technical working group Wear Test Methods (TWA 1) has initiated projects to stimulate the introduction of advanced materials, such as wear resistant ceramics and inorganic coatings, into high technology products and engineering structures through international agreement on test methods and performance standards. Although there exists a strong conviction among the industrialized countries that advanced materials are the roots of technological growth, the introduction of new or improved materials for contemporary industrial applications is often hindered by the lack of reliable data and test methods for these materials. This is especially true with respect to materials for tribological applications because of the complexity of the friction and wear processes and the lack of standardized test methods.

Consequently, the working group Wear Test Methods started a round robin exercise to establish the reproducibility of sliding friction and wear tests in support of developing internationally accepted wear test methodologies, which should lead to an improvement of reproducibility and comparability of tribological test results.

2 First Round Robin Study

The target of this first international round robin exercise was the characterization of wear behavior of a selected ceramic in comparison with a conventional material. The test system was:

- stationary ball with 10 mm diameter, materials Al_2O_3 and AISI 52100 steel; continuous sliding against rotating disk, track diameter 32 mm, materials Al_2O_3 and AISI 52100 steel;

- sliding velocity 0.1 m/s; normal load 10 N; temperature 23 °C ±1 °C; sliding distance 1000 m

- pairings: steel ball against steel disk, alumina ball against steel disk, steel ball against alumina disk, alumina ball against alumina disk.

A total of 32 laboratories from 7 countries participated in this comparison. All participants were supplied with identical specimen couples (geometry, material composition, mechanical and surface properties). A defined specimen-cleaning preparation prior to each test was also recommended. The results were published by H. Czichos, S. Becker and J. Lexow in Wear, **114** (1987) 109-130.

3 Second Round Robin Study

In the second round of VAMAS multilaboratory comparisons, the same test conditions as defined for the first round were applied. An additional material, Si_3N_4, was studied, giving five different material pairings (Si_3N_4/Si_3N_4; Si_3N_4/AISI 52100; Si_3N_4/Al_2O_3; AISI 52100/Si_3N_4; Al_2O_3/Si_3N_4).

A total of 38 participants from 9 countries contributed results to this second round, which were published by H. Czichos, S. Becker and J. Lexow in Wear, **135** (1989) 171-191. The following conclusions can be drawn from both rounds.

(1) The first and the second round of VAMAS intercomparison led to the same range of wear and friction values for the steel/steel pairings thus proving the reproducibility of wear tests for this reference material pairing.

(2) The system wear (W_1, total linear wear) of similar material pairings of different types was significantly different; the following ranking order was observed:

$$W_1(Al_2O_3\text{-}Al_2O_3) < W_1(Si_3N_4\text{-}Si_3N_4) < W_1(\text{steel-steel}).$$

(3) The system wear of all dissimilar material pairings was significantly different in the following ranking order:

$W_1(Al_2O_3\text{-Steel}) < W_1(\text{Steel-}Si_3N_4) < W_1(Si_3N_4\text{-}Al_2O_3) <$

$W_1(Si_3N_4\text{-Steel}) < W_1(\text{Steel-}Al_2O_3)$.

(4) For steel-ceramic pairings, an exchange of the ball and disk material led to significantly different values:

$W_1(Al_2O_3\text{-Steel}) < W_1(\text{Steel-}Al_2O_3)$

$W_1(Si_3N_4\text{-Steel}) > W_1(\text{Steel-}Si_3N_4)$.

(5) The friction coefficients (f) of similar material pairings of different type is significantly different; the following ranking order was observed:

$f(Al_2O_3\text{-}Al_2O_3) < f(\text{Steel-Steel}) < f(Si_3N_4\text{-}Si_3N_4)$.

$\sim 0.4 \pm 0.08 < 0.6 \pm 0.1 < 0.7 \pm 0.2$.

(6) The reproducibility of friction data (interlaboratory) was in the range ±18% to ±20%. The reproducibility of system wear data (interlaboratory) was in the range ±29% to ±38%.

Based on these results, a DIN and an ASTM standard (DIN 50324, ASTM G-99) were developed and adopted.

4 Current Work Program

Four projects are currently in progress.

(1) Standard format for organizing and reporting wear data (coordinated by Dr. A. W. Ruff, NIST, USA).

(2) Interlaboratory wear volume measurement comparison (coordinated by Dr. M. Gee, NPL, UK).

(3) Compilation of standard wear test methods (coordinated by Dr. P. Blau, ORNL, USA).

(4) Interlaboratory wear testing of inorganic coatings (coordinated by Dr. E. Santner, BAM, Germany).

The objective of project No. 1 is the definition of a uniform format for organizing and reporting wear data with application to computerized databases. This project is important and timely, because of the growing interest in using computer databases for storing materials property data and searching the databases to retrieve data for research, design and material selection. There exist large differences between data report formats in different organizations and countries. If a uniform format can be developed, then it will be much easier to exchange and compare reported data. Other benefits of this project may be an agreement on important variables and units, as well as methods for reporting data precision. Completion is expected in 1994.

The goal of project No. 2 is to evaluate and compare different methods used for wear volume measurements. The results will be information on the uncertainty that can be expected from different methods of measuring and calculating the wear volume. Because the measurements will be done on exactly the same samples, the resulting deviations of wear volume are pure uncertainties from measurement of this quantity and not influenced by inhomogeneities of test specimens and deviations of test parameters and test rig constructions. Completion is expected in 1994.

The purpose of project No. 3 is to gather and disseminate a listing of currently available standards for wear testing in all participating countries, excluding pure industry standards. This activity would serve as an education document and as a resource to help in developing wear testing standards throughout the world. Publication of the compilation as a VAMAS report is expected in 1993.

For project No. 4, "Interlaboratory wear testing of inorganic coatings," the test specimens will be disseminated to the participating laboratories after characterization. For the tests, a TiN-coating has been chosen because of its widespread use in practical applications and good production reproducibility. With one possible exception, the test parameters will be the same as those in the two former round robin exercises. Only the sliding distance may be changed for the tests with the 7 µm thick coatings. This project is sponsored by FAG-Kugelfischer, Georg Schäfer KGaA, supplying the steel balls made from high temperature bearing steel M 50, and by Deutsche Balzer GmbH preparing the TiN-coating on balls and disks. High temperature steel M 50 was chosen as the substrate material to avoid hardness decrease during the coating process. Completion of project is expected at the end of 1994.

Technical Working Area 2

SURFACE CHEMICAL ANALYSIS

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Objectives

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

During this period we have seen some very positive progress with some projects now close to completion and others moving more rapidly as shown in Figure 1. Project status is summarized under the five theme areas; (i) basic theory of surface analysis, (ii) calibration of instruments; (iii) development and validation of software and formats; (iv) reference materials; and (v) reference data. A natural consequence of the genuine need for work and standards in this area has been the recent establishment of Technical Committee 201 on Surface Chemical Analysis by the International Standards Organization. This is detailed in the separate article in this issue by C. J. Powell and R. Shimizu. TWA 2 now has liaison category A with ISO TC/201 which ensures full communication between the two activities so that the results of any VAMAS work may rapidly enter the TC/201 activity.

The basic theory studies concern the overall spectral shape as a function of the angle of emission of the electrons in Auger Electron Spectroscopy (AES) and X-Ray Photoelectron Spectroscopy (XPS). From this knowledge, the elements present can be determined. By removing the true spectral shape of the background, the true spectral intensity of the peaks can be obtained and hence quantify the analysis. By understanding either the true background shape or the angular dependence of the emitted spectral peak intensities, we may deduce the depth distribution of the observed elements quantitatively. In order to do this, calibrated spectrometers are needed to obtain true spectral intensities and validate software algorithms to ensure that they do what they are supposed to. This validation in turn requires reference materials and data. When this TWA started none of these problems had been resolved and clear ideas of how to solve them had not been formulated. As each item in the work programme has been resolved its results have been taken up by the next project to move onwards. The basic theory studies concerning the overall spectral shape in electron spectroscopy resides in projects 7 and 14. Results in project 14 are reaching a successful conclusion in convergence with data recorded on instruments calibrated via projects 9 and 23. These calibration systems have been proven and are to be established on a more formal basis. Project 7 was progressing very well indeed but has stalled through lack of manpower. A transfer of the work to another institute is being sought. Projects 14 and 26 feed into the angle-resolved AES and XPS measurements of projects 3 and 31 to give depth distribution profiles of the elements measured over the outermost 6 nm of the surface zone. Project 3 has developed very strongly and it may soon become necessary to consider if the original objectives need to be broadened. In project 2, the long awaited NIST values for the Al and Mg $K\alpha_{12}$ centroid energy values are now available and these give us greater confidence in our ability to assign Auger energy values.

To assist in the transfer of data from laboratory to laboratory a Standard Data Transfer Format was developed in project 10. This is now in use in Japan, the UK and the USA and is being implemented by instrument manufacturers. To assist this, a file format translation system was developed in project 29 which then allows data to be input to the Common Data Processing Test-bed System developed in project 30. Project 30 allows software to be developed to address certain activities without the need to write all of the other ancillary data handling and processing activities each time. Complementary to this approach is the development of test objects to inject into commercial processing systems to test the validity of their software. In project 21 a structure with known noise and known constituent peaks can be injected into software receiving the standard format to perform such tests. This project may be expanded to provide tests in commercial formats.

An integral part of quantification and analysis is the development of reference materials. These also serve to test if the analytical procedure is working properly. Many analysts are interested in materials which are insulators. Project 6 is moving through a pre-round robin test to set up polymer materials for calibrating neutralizing systems in XPS instruments, learning about analyzing polymers and their quantification. Although materials have been supplied, the return of results is rather slow. Project 11 is setting up protocols and understanding of charging in insulators in general. Project 12 allows surface layers to be quantified by a surface sensitive but not surface specific nuclear beam technique. This is accurately quantitative and can be used in the future to calibrate relevant transfer standards. Transfer standard reference materials are much easier to prepare for composition depth profiling than for surface analysis as the surface contamination resulting from storage and transit is largely unimportant. Projects 5 and 19 are now moving forward more strongly in the development of reference materials for Secondary Ion Mass Spectrometry (SIMS) and project 15 for Sputtered Neutral Mass Spectrometry (SNMS) has linked to a similar activity in Germany. Reference materials for studying the oxide sputtering yields for depth profiling (project 8) and for chemical state in XPS are now being assisted by work being started in ISPRA in Italy.

This brief survey does not cover all of the projects but highlights the points where new developments are occurring. It is clear that the more mature techniques of AES and XPS are very effectively covered here whereas the newer techniques of static SIMS and SNMS are poorly covered. These techniques have greater potential than AES and XPS but have much more severe problems. Because of their sensitivity and detailed specification they can address problems that AES and XPS cannot address and hence their problems require urgent solution. It is hoped to focus more effort in this area in the future.

Details of projects will be found in the full reports distributed by National Representatives. The distribution of project leadership, as shown below, demonstrates a broad-based interest in this VAMAS activity:

Canada	=	12, 13
France	=	7, 11, 16, 31
Germany	=	5, 8
Italy	=	15
Japan	=	17, 19, 20, 25, 26, 28, 29, 30
UK	=	1, 2, 9, 10, 21, 22, 23, 27
USA	=	2, 3, 6, 18, 24
CEC	=	14

VAMAS SCA PROJECT LIST (1 DECEMBER 1992)

Project No:

1.* Development of thin oxide films as reference materials (M. P. Seah).
2.* Development of calibration data for the energy scales of Auger-electron spectrometers (M. P. Seah, C. J. Powell).
3. Procedures for quantitative X-ray photoelectron spectroscopy (C. J. Powell, J. E. Fulghum for Phase 1).
4. Measurement of spatial resolution in AES.
5.* Development of reference materials prepared by ion implantation (W. H. Gries, D. Gould).
6. XPS intensity calibration and stabilization with polymeric reference materials (C. E. Bryson).
7. Correction methods for backscattering in AES (J. P. Langeron).
8.* Reference data for sputtering rates in oxides (H. J. Grabke).
9.* Intercomparison of Auger-electron energy and intensity measurements (M. P. Seah).
10. Development of a standard data transfer format (W. A. Dench).
11.* Multitechnique characterization of vacancies in alumina (C. Le Gressus).

12. Calibration of surface layers by nuclear reaction analysis (I. V. Mitchell).
13. Tests of algorithms for data processing AES - Factor analysis and intensity (P. R. Underhill).
14.* (a) Tests of algorithms for background subtraction in XPS (S. Tougaard).
(b) Tests of algorithms for background subtraction in AES (S. Tougaard).
(c) Tests of algorithms for quantitative XPS by peak and peak background shape analysis (S. Tougaard).
15. Evaluation of SNMS sensitivity factors (M. Anderle).
16.* Intercomparison of surface analysis of thin aluminum oxide films (P. Marcus).
17. Quantitative AES of Au/Cu alloys (R. Shimizu).
18. Evaluation of LOGIT, an algorithm for fitting sputter-depth-profile data, for the measurement of interface widths of an NBS thin-film reference material (J. Fine).
19. Round Robin SIMS study of impurities in Ga/As crystals (S. Kurosawa).
20. Round Robin AES study of Co-Ni alloys (K. Yoshihara).
21. Tests of algorithms for the analysis of multicomponent spectra in XPS (A. F. Carley).
22. Calibration of channel electron multiplier detection efficiency stabilities (M. P. Seah).
23.* Absolute calibration of XPS instrument intensity scales (M. P. Seah).
24. Conventions for spectral databases (R. N. Lee).
25. Quantitative XPS of Au-Cu alloys (K. Yoshihara).
26. Theoretical assessment of escape depth (R. Shimizu).
27.* Multiline reference material for differential AES intensity calibration (M. P. Seah).
28. Quantitative XPS of Co-Ni alloys (K. Yoshihara).
29. Development of a File Format Translation System (K. Yoshihara).
30. Development of a Common Data Processing System for AES and XPS (K. Yoshihara).
31.* Intercomparison of the Effects of Al on the Determination of Thin Oxide Film Thicknesses (P. Marcus).

* these projects receive support from the Community Bureau of Reference (BCR), Brussels

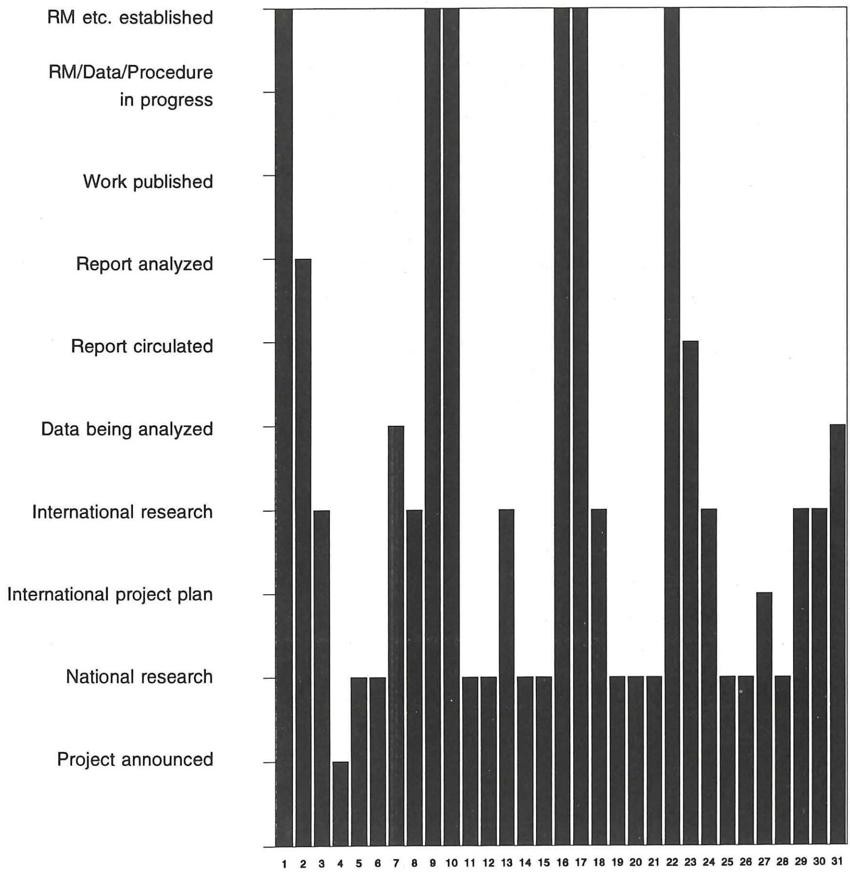

Figure 1. TWA2 Project Status Chart (December 1992)

CERAMICS

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Objectives

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

There has been substantial progress on several programs that are simultaneously under way.

The room-temperature fracture toughness round robin organized by the Japan Fine Ceramic Center (JFCC) in 1989 has now concluded. Partial results (thirteen of twenty-two participating results) had previously been published as VAMAS Technical Report No 9, and in condensed form in the Journal of the Ceramic Society of Japan, International Edition. Five USA labs completed their work in 1992 and a report with these results as well as the earlier 13 was published in the NIST Journal of Research in late 1992. Consistent results were obtained in this study with the Indentation Strength (IS) and Single-Edged Precracked Beam (SEPB) methods. Less consistent results were obtained with the Indentation Fracture method.

Two of the three test methods used in the round robin are part of a Japanese standard for fracture toughness evaluation: JIS R 1607. Follow-on studies are now under way in Europe (a European Structural Integrity Society round robin) and in the USA (ASTM committees C28, Advanced Ceramics, and E8 Fracture and Fatigue).

The high-temperature fracture toughness round robin, also coordinated by JFCC, is continuing and should be complete in early 1993. Single-edged precracked beam, single-edged V notched beam, and chevron notch specimens are being tested.

The activities of this Working Area expanded beyond mechanical testing in 1992 to include new work on quantitative microscopy. A planned CEN round robin with fourteen European labs was expanded to become a joint CEN/VAMAS project with six USA and four Japanese laboratories. The Centre for Technical Ceramics in the Netherlands and the National Physical Laboratory in the United Kingdom coordinated this project. Average grain size, and grain size distribution was evaluated for two aluminas and a computer generated microstructure. Participants analyzed furnished photos and also prepared specimens themselves. There was no statistically significant difference between results obtained with the line or circle manual counting methods, provided that a minimum number of intersections was used. In general, the random placement of lines or circles on a given photograph and the interpretation of the intersections led to a 10% variation in results. Additional variability, of the order of 15% more, was incurred when participants prepared their own micrographs.

The quantitative microscopy round robin was conceived, set up, conducted, analyzed, and a final report written in less than one year. An extended abstract of this report is found elsewhere in this Bulletin. All participants felt that the project was a success and was very worthwhile. The results were used to prepare the new Euronorm: ENV 623 Determination of Grain Size for Advanced Technical Ceramics.

Finally, a new round robin was initiated in late 1992 on fracture toughness of advanced ceramics by the controlled surface flaw method. This program is coordinated by the National Institute of Standards and Technology, USA and the Swiss Federal Laboratories for Materials Testing and Research. Seven hundred and twenty silicon nitride and zirconia specimens were distributed in November 1992.

A new project on fractographic analysis of ceramic specimens is being planned.

Technical Working Area 6

SUPERCONDUCTING AND CRYOGENIC STRUCTURAL MATERIALS

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Objectives

- To establish reliable measurement techniques for superconducting materials, initially through the use of round robins on critical current and AC loss measurements in multifilamentary wires
- To establish reliable measurement techniques for cryogenic structural materials, initially through the use of round robins on tensile and fracture toughness measurements

In 1992, the TWA met twice, - Wien in June and in Chicago in August; each meeting had 18 participants. The report on the intercomparison of the critical current measurement in Nb_3Sn wires has been summarized following the discussion at the TWA meetings. The report includes background techniques for critical current measurement, results of the 1st and 2nd intercomparisons, and a recommended method of critical current measurement in Nb_3Sn wires. The report will be submitted to *CRYOGENICS* soon, and effectively transferred to the activities of IEC TC-90.

The results of the intercomparison on the AC loss measurement in Nb-Ti wires based on the AC magnetization method was summarized at the June TWA meeting, and that based on the VSM method was summarized at the August TWA meeting. Another supplemental intercomparison using the VSM method is being arranged. A new intercomparison program on the critical field measurement in Nb-Ti wire was proposed at the August meeting, and initiated in January 1993. The measurements will be completed by the end of June 1993. The number of participant laboratories in this new intercomparison is eleven.

Meanwhile, intercomparisons of the tensile and fracture toughness tests of cryogenic structural materials at 4.2 K have been performed. The tensile results show good agreement under the improved testing conditions and requirements. In the fracture toughness test, some of the testing variables are still under discussion, although better agreement has been obtained in the 2nd intercomparison. The report on the tensile and fracture toughness intercomparisons is being prepared, and will be published in *CRYOGENICS* by the end of this year. In another intercomparison program of strain gauge calibration at cryogenic temperatures, the apparent strain and gauge factor measurements have been recently completed. The results are being summarized and analyzed.

MATERIALS DATABANKS

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Objectives

- To assess the role of standards in the flow of computerized materials information
- To identify needs, problem areas and options for standardization activity and coordinate pre-standardization research

TWA 10 activities have so far proceeded in three phases: Phase 1 - a survey-oriented phase which was concluded in 1987; Phase 2 - preparatory and pre-standardization activities, and Phase 3 - definition of two pre-standardization projects.

Phase 1

- Consensus Report of a VAMAS Working Group: Factual Materials Databanks - The Need for Standards (VAMAS Technical Report No. 2, July 1987).

Phase 2

- VAMAS Workshop on Standards for Materials Databanks, JRC-Petten, November 1988, (VAMAS Technical Report No. 4, November 1989).

- International Workshop on Materials Data Interchange, Rolls-Royce plc, Derby, September 1989 (Follow-up meeting of the Data Interchange, Group of the VAMAS Workshop at Petten).

- Inventory of Materials Designation Systems, characterizing features and structures of all known materials designation systems world-wide. Report by K. W. Reynard, UK, to be published.

- Interlaboratory Comparison of computerized Data Evaluation Methods, organized and evaluated by NRIM, Tokyo. Fourteen participating institutions; final report (in two parts) by S. Nishijima, Y. Monma and K. Kanazawa, NRIM, (VAMAS Technical Reports No. 6 and 7).

Phase 3

- Inventory of Methods/Models for Materials Data Analysis and Evaluation. Activity led by the National Research Institute of Metals (NRIM) at Tokyo with the New Materials Centre (NMC) at Osaka as the secretariat. Twenty-four institutions in VAMAS countries and China, Czechoslovakia, Poland and USSR, supplemented by many additional proposals for candidate participation. The work not only addresses metals and alloys but also polymers and ceramics. A draft report of the initial phase is available.

- Materials Data Interchange. It was planned to compare Materials Data Interchange Methods in a number of inter-institutional data exchange tests. ASTM Committee E49 would produce a first round of data exchange tests preceding the VAMAS activity. These experiments were abandoned because it had turned out that the required programming efforts were excessive. Simultaneously the STEP (Standards for the Exchange of Product Data) activities have made significant progress in the development of a materials model which has the potential to provide a basis for implementing a universal material data exchange method. All related activities world-wide including ASTM-E49 are now focused on the STEP approach. At present, TWA10 cannot contribute to this new development. It is therefore proposed to suspend work in this until progress in the STEP materials development will allow the start of pre-normative validation testing which should be organized under the VAMAS aegis.

Technical Working Area 14

THE TECHNICAL BASIS FOR A UNIFIED CLASSIFICATION SYSTEM FOR ADVANCED CERAMICS

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Objectives

- To identify and assess issues inherent in the development of an internal classification system for advanced ceramics, particularly terminology and nomenclature.
- To establish a suitable classification structure and mechanisms for system implementation.

TWA 14 has reached a significant milestone in that the first phase of planned accomplishments has been achieved. These culminated in the formulation of a full range classification system for advanced ceramics. The development of this classification scheme was made possible through several inter-related work activities, including:

- The completion of an international survey of 120 industrial organizations to ascertain their current classification practices and to determine guidance on their preferred schemes. A VAMAS report describing the survey was issued in May 1991 (ISSN 1016-2186).

- An international workshop was organized in June 1990 to develop the primary elements (the building blocks) of an industrial preferred system, using criteria set from analysis of the survey. The workshop proceedings will be published in the near term by Elsevier Science Publishers, Ltd in their "Ceramics International" publication.

- The implementation of a special project to develop in full a classification system responsive to the directions evident from the two previous activities. This project was conducted for TWA 14 by a private consultant, Ceram Research. The consultant's work, sponsored by the Community Bureau of Reference, the USA

(Institute for Standards Research/ASTM) and Japan (Japanese Fine Ceramics Association), and that of TWA 14 membership, resulted in the formulation of a matrix classification system having the following primary descriptor fields; Application, Chemical Character, Processing and Property Data.

A complete report describing the background, actions and developed classification system will be published in 1993 under the auspices of VAMAS and made available to all interested standards and other organizations.

A second phase of TWA 14 activities will be initiated to test the classification system and its coding scheme in detail, using developed software and a variety of databases.

• VAMAS Calendar •

TWA 4 Meeting UMIST, Manchester, UK	April 5, 1993
TWA 1 Meeting San Francisco, CA, USA	April 13-16, 1993
TWA 3 Meeting Fraunhofer Institute, Freiburg, Germany	May 13, 1993
TWA 6 Meeting Albuquerque, NM, USA	July 12, 1993
TWA 15 Meeting Metal Matrix Composites, Madrid, Spain	July 14, 1993
TWA 3 Meeting Japanese Fine Ceramics Center, Nagoya, Japan	August 1993
ASTM E49 Meeting NIST, Gaithersburg, MD, USA	October 4-5, 1993
4th International Symposium on the Computerization of Materials Databases, NIST, Gaithersburg, MD, USA	October 6-8, 1993
CODATA Task Group on Material Database Management NIST, Gaithersburg, MD, USA	October 8-9, 1993
TWA 3 Meeting ASTM/ACerS Meeting, Cocoa Beach, FL, USA	January 1994
8th Cimtec Meeting, *Forum on New Materials* & *World Ceramics Congress* Florence Congress Center, Florence, Italy	June 29 - July 4, 1994

• VAMAS Calendar •

EIGHTH CIMTEC INTERNATIONAL CONFERENCE

Authorized by the VAMAS Steering Committee, VAMAS is one of the organizations officially endorsing the 8th Cimtec and its two major themes, the *Forum on New Materials* and the *World Ceramics Congress*. The VAMAS Chairman serves on the Presidential Committee of the 8th Cimtec. This international conference will be held June 29 to July 4, 1994 at the Florence Congress Center, Florence Italy. The Call for Papers for both the Forum on New Materials and the World Ceramics Congress have been issued. Contributed papers for oral and poster sessions will be selected on the basis of submitted abstracts. Abstracts and appropriate application forms must be submitted in English by October 15, 1993 to the Congress Secretariat.

The Forum on New Materials program will consist of: a General Session `New Horizons in Materials Research' dedicated to fundamental aspects of process science and advanced characterization of materials; eight Topical Symposia on applications-driven R&D areas (jointly sponsored with the World Ceramics Congress); and a Special Session `Standards and Markets for Advanced Materials'. The eight Topical Symposia include: inorganic films and coatings; diamond and diamond-like films; structural fiber composites; superconducting materials; high performance materials in engine technology; intelligent materials & systems; advanced materials for optics, electro-optics and communication technologies; and materials in clinical applications.

The World Ceramics Congress program focuses on Classical Ceramics (10 sessions) and Advanced Technical Ceramics (14 sessions). Two international Symposia will be held on Ceramics in Architecture and The Ceramics Heritage.

For more information, write to 8th Cimtec, P.O. Box 174, 48018, Faenza, Italy.
Telephone: +546-22461 or +546-664143
Fax: +546-664138 or +546-663362

• Participants & Publications Update •

Each VAMAS participant should have received a copy of the revised Participants and Publications document dated May 1992. This document contains two sections: membership listings for the VAMAS Secretariat, VAMAS Steering Committee, Steering Committee Contacts, Technical Working Areas (TWAs), ISO Contacts, and interested parties who receive the Bulletin but who are not active participants; and the associated VAMAS publications from the TWAs.

It is anticipated that this publication will be re-issued in late 1994. In order to produce this comprehensive summary as expeditiously as possible with the most up-to-date information, the TWA Chairs and other individuals are strongly encouraged to review the individual entries and the publications list and report all changes to the VAMAS Secretary. All corrections, additions, and deletions to the membership lists should be reported in the following format:

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TITLE:
ORGANIZATION NAME:
STREET ADDRESS:
CITY, STATE OR PROVINCE:
COUNTRY, POSTAL CODE:
TELEPHONE NUMBER: [INCL. CITY AND COUNTRY CODES]
FAX NUMBER:
TELEX NUMBER:

All corrections and additions to the publications list should contain the following:

AUTHORS:
TITLE:
REPORT OR JOURNAL TITLE:
REPORT NUMBER OR VOLUME NUMBER:
BEGINNING JOURNAL PAGE NUMBER:
PUBLICATION YEAR:

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