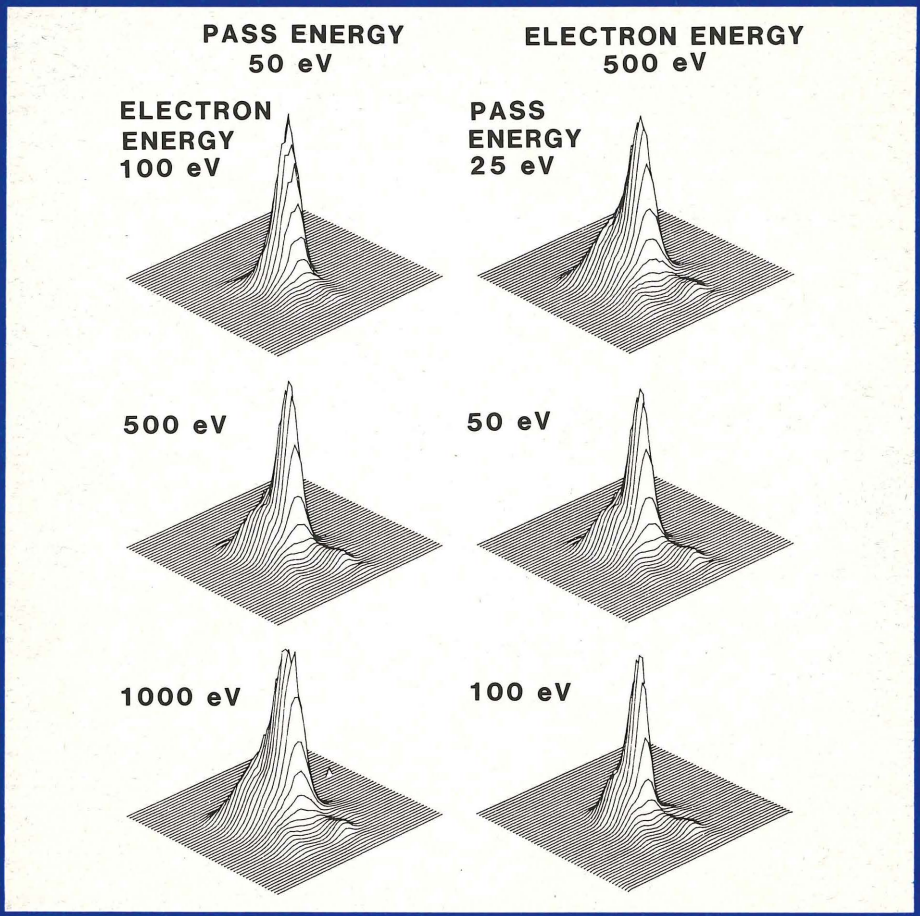
VAMAS



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Versailles Project on Advanced Materials and Standards
Canada France Germany (FRG) Italy Japan UK USA CEC

VAMAS

• MILESTONES •

VAMAS has recently passed two extremely important milestones that call for comment. The first is the completion of the process of definition, approval, and signature by all eight governments of the Memorandum of Understanding under which it is now working. Founded as one of eighteen international cooperative projects at the Economic Summit of Heads of State five years ago in Versailles as a part of the framework of that series of meetings, VAMAS addressed such a clear need for a joint international effort in prestandards research that steps to provide permanence and independence for its operation soon began.

We are indebted to the French for the concept and birth of the eighteen projects on Technology, Growth, and Employment started at Versailles. We are indebted to the United Kingdom for the realization of the importance of international cooperation on the prestandards research on advanced materials within this framework. To the United Kingdom, we are indebted also for leadership during the crucial first three years of existence, the honing of the Memorandum of Understanding as part of its concept of this leadership, and the achievement of agreement on the Memorandum among all eight governments of the Economic Summits.

VAMAS has clearly been a growing, organic body since its inception, with a variety of complementary contributions to its development by all eight participants. As VAMAS grows, it is contributing in a number of important ways to the achievement of international understanding, both scientific and institutional, and to the avoidance of unfortunate barriers to trade involving high technology materials. Completion of the Memorandum of Understanding underlines the importance that the eight governments continue to attach to these goals. The vitality of the organization underlines the importance that individual scientists and engineers attach to prestandards research and the effectiveness of the consensus process in achieving international collaboration.

The second milestone that VAMAS has passed is the completion of its first technical project, including journal publication of the results. This involves an intercomparison on the wear of steel and ceramics in various combinations. The project, led by the Federal Republic of Germany, is widely regarded as a model for one of the most important types of VAMAS activity. Several results are immediately apparent. Among these are two new VAMAS publications, a series of VAMAS Reports and a series of VAMAS Technical Notes, which will take their place along with journal publication as major products for VAMAS. Those reports, suitable for wide distribution, will appear as Supplements to the VAMAS Bulletin. The Technical Notes, which will contain a summary of the most important information from the Reports and journal publications, will be published in the VAMAS Bulletin. Moreover, the scientific results are already being incorporated in standards being drafted by national standards bodies.

Thus immediately upon the completion of the Memorandum of Understanding, VAMAS is already showing a variety of results. Indeed it is fulfilling its promise to accelerate the development of standards on advanced materials.

Cover: Images recorded with an Auger-electron spectrometer to show the specimen areas contributing to the detected signal for the indicated conditions of analyzer pass energy and electron kinetic energy.

PROGRESS OF THE VAMAS SURFACE CHEMICAL ANALYSIS WORKING PARTY

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INTRODUCTION

The Surface Chemical Analysis Working Party was one of the first cooperative projects to be initiated under VAMAS. This article summarizes accomplishments and describes the scope of work being conducted by the Working Party.

CONTEXT OF ACTIVITY

Surface analysis is now in widespread use for a great variety of scientific and technological purposes, particularly in the development of advanced materials. Examples of current applications are: the development of advanced microelectronic materials and devices (where surface processing is critical); the development of surface treatments for metals, such as ion implantation, to improve their mechanical and wear properties; the development of advanced polymer materials; investigations of adhesion at the interfaces of composite and thin-film materials; and the development of improved coatings to minimize corrosion.

METHODS OF SURFACE ANALYSIS

Three surface analysis techniques are in widespread use.[1] These techniques are Auger-electron spectroscopy (AES); x-ray photoelectron spectroscopy (XPS), sometimes referred to as electron spectroscopy for chemical analysis (ESCA); and secondary-ion mass spectroscopy (SIMS). The specimen material is excited by electrons (AES), x-rays (XPS), or ions (SIMS); either electrons (AES and XPS) or ions (SIMS) are detected. Measurement of characteristic electron energies (AES and XPS) allows elemental identification to be made (except for H and He); and either small shifts of these observed energies or the spectral lineshapes can often be used to identify the chemical states of the elements. Mass analysis of emitted ions (SIMS) similarly allows identification of elements and compounds.

The three surface analysis methods can each be utilized in several different operating modes. First, an analysis can be made of a *particular region* on a specimen surface. The best lateral resolution of commercial instruments (in the plane of the surface) varies and can range from about 50 nm for AES and about 200 nm for SIMS to about 250 μm for XPS. Research instruments can do somewhat better. The best depth resolution varies between about 0.5 nm and 3 nm for AES and XPS, depending on element and specimen material, and between about 0.5 nm and 10 nm for SIMS, depending on whether the selected lateral resolution is low ($\approx$ 1 mm) or as high as that quoted earlier.

In the second operating mode, the *distribution* of elements in a thin layer parallel to the surface can be obtained from spectra measured when a narrow incident electron (AES) or ion (SIMS) beam is deflected to selected regions on the surface in a sequential manner. Elemental distributions can also be obtained with a broad x-ray (XPS) or ion (SIMS) beam incident on the surface and with the analyzer system operating in a microscope mode.

Finally, the composition *normal* to the specimen surface can be determined by measuring the surface composition (at one or more points) while surface material is removed by ion bombardment. A sputter-depth profile (SDP) is obtained by plotting elemental concentrations versus depth from the original surface.

This brief synopsis overlooks other important considerations.[1] The sensitivity for elemental detection with SIMS can be at least several orders of magnitude better than for AES or XPS, but depends greatly on chemical state and the charge of the incident and emitted ions. A relatively recent development, sputtered-neutral mass spectroscopy (SNMS), promises a considerable reduction in the large variations of SIMS elemental sensitivities with matrix. For all methods, sensitivity is reduced as the spatial resolution is improved. Attempts to improve spatial resolution cannot always be realized since some materials will decompose before useful data can be acquired.

NEED FOR STANDARDS AND INSTITUTIONAL RESPONSE

The above-mentioned techniques for surface analysis are conceptually simple. Nevertheless, operation of the instruments is complex. There are many options in the design of experiments and the acquisition of data, and there are many uncertainties and limitations in present methods for data reduction and analysis. Reference procedures are needed for users to avoid the many artifacts that can occur during instrument operation and to enable efficient and reliable processing of spectral data. Reference data and reference materials are needed for the calibration of instrumental scales (e.g., energy and intensity scales for AES and XPS, and depth scales in SDP). Reference data are required for elemental sensitivity factors, the many parameters that influence observed spectral lineshapes and intensities, ion sputtering rates in materials, and the extent to which ion bombardment modifies the surface composition and topography.

These and other needs for surface analysis standards have been documented previously.[2,3] Awareness of these needs has led over the past ten years to increasingly active programs at the U.S. National Bureau of Standards (NBS) and the U.K. National Physical Laboratory (NPL). The Community Bureau of Reference (BCR) of the Commission of the European Communities (CEC) has provided important support for European effort.

ASTM formed Committee E-42 on Surface Analysis in 1976 to initiate the process of developing formal documentary standards or reference procedures.[3,4] The Committee has now issued thirteen such standards with over ten proposed standards under consideration in the ASTM review and approval system.[5] An important aspect of the Committee's work has been the organization of round robins or interlaboratory comparisons in which the results of nominally identical measurements were compared. In a number of the round robins, disparities in the results have clearly indicated the need for improved reference data and for relevant reference procedures. The

Committee also has sponsored symposia and workshops on surface-analysis topics and has been a focus for discussion on needs for reference materials and reference data.

The ASTM E-42 Committee has made a useful start in the development of standards for surface analysis. In the past few years, a Working Group on Surface Analysis (WGSA) of the International Union of Pure and Applied Chemistry (IUPAC) Commission V.2 on Microchemical Techniques and Trace Analysis has drafted several documents that give recommendations for the surface analysis of semiconductors; additional documents are in preparation. These efforts to develop surface analysis standards have been and continue to be very useful. Nevertheless, it has become apparent that the quality and reliability of many surface analyses are not as high as they could and should be, and that users of surface analysis instrumentation are suffering frustration due to the lack of needed reference procedures, reference data, and reference materials. These concerns have intensified with the recent growth of applications involving surface analysis.

THE VAMAS SURFACE CHEMICAL ANALYSIS WORKING PARTY

The formation of VAMAS came at an opportune time for surface analysis. The need for research leading to the standards required for surface analysis is much greater than can be satisfied rapidly by current programs at NPL and NBS and by the efforts alone of groups such as the ASTM E-42 Committee and the IUPAC WGSA.

All of the VAMAS states and the CEC are participants in the Surface Chemical Analysis Working Party (SCAWP). The objective of the SCAWP is to produce by coordinated effort the reference procedures, reference data, and reference materials necessary for the various standards organizations to establish standards for surface analysis.[6,7]

Formal meetings of the SCAWP national representatives were held in October, 1985, and November, 1986, at which a number of cooperative activities were approved. Brief descriptions are given below of two initial activities together with a synopsis of other cooperative activities that have recently started. Information is also given on some national activities and on meetings co-sponsored by the SCAWP.[7]

a. Development of Thin Oxide Films as Reference Materials (M.P. Seah, UK)

NPL and CEC BCR have recently produced a tantalum oxide on tantalum material (NPL No. S7B83; BCR No. 261). This material consists of four rectangular foils, 5 x 10 mm, oxidized on both sides, in each of two oxide thicknesses (nominally 30 nm and 100 nm).[8] The metal-oxide interface is particularly sharp and this property makes the material useful for optimizing instrumental conditions in SDP measurements.

The VAMAS project involved a collaboration between scientists at NPL, Chalk River, and McMaster University (Canada), Université Paris VII, and Université de Compiègne (France), Université de Liège (Belgium), Harwell, and University of Surrey (UK), with CEC support.[9] Comparisons were made of the anodically grown tantalum oxide films produced independently at NPL, Chalk River, and Paris from which it was found that the material from each laboratory had the same composition and showed a constant sputtering rate with depth (for 2 keV argon ions).

In addition, the thicknesses of the NPL/BCR oxide films were established using several techniques available in the participating laboratories: charge transfer during film growth, optical reflectometry, and nuclear reaction analysis. These measurements yielded oxide thicknesses of $1.79 \pm 0.04 \times 10^{21}$ and $5.40 \pm 0.10 \times 10^{21}$ oxygen atoms/m^2 for the thin and thick oxide films, respectively. As a result of this further characterization, the NPL/BCR material can be used for the measurement of ion flux density, calibration of Faraday cups, and calibration of nuclear scattering experiments.

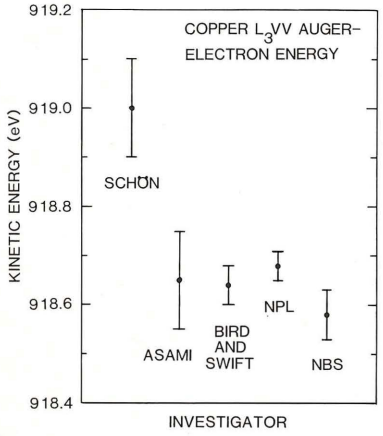

FIGURE 1. Reported values and uncertainties for the kinetic energy of the copper L_3VV Auger peak relative to the Fermi level.[12] The NBS value is a preliminary result.

b. Development of Calibration Data for the Energy Scales of Auger-Electron Spectrometers (M. P. Seah, UK, CEC Support; C.J. Powell, USA)

Accurate measurement of Auger-electron kinetic energies is important for the reliable determination of the chemical states of surface elements. An ASTM E-42 round robin in which comparisons were made of measured energies of Auger electrons from pure copper and gold showed a spread ranging from 7 eV for a Cu peak nominally at 60 eV to 32 eV for gold peak nominally at 2025 eV.[10] Spreads of this magnitude preclude reliable chemical-state determination and demonstrate the need for reference data to calibrate the energy scales of AES instruments.

Several reports have been published in which Auger-electron energies are given to reasonably high accuracy (as indicated by the data for a copper Auger peak shown in Fig. 1). NPL and NBS each decided that new measurements of selected Auger-electron energies traceable to national standards of the volt should be made. Independent measurements have been made of Auger-electron energies for Cu, Ag, and Au which are suitable reference materials. Preliminary results for the Cu L_3VV Auger peak are shown in Fig. 1. When all measurements have been completed, a careful comparison will be made of the NPL and NBS results. If the results are consistent (within the measurement uncertainties), joint recommendations will be made of values to be used for instrument calibrations.

NPL and NBS each had to perform a sequence of careful measurements to characterize the instruments used in their AES calibration experiments. The front cover shows elastic-peak "images" that demonstrate how the NBS analyzer

viewed different specimen areas depending on the selected operating parameters.[11] The widths of these images are in partial agreement with design data, the deviations (particularly the asymmetries) being due in large part to manufacturing tolerances. A standard based on NPL and NBS techniques for measuring the active specimen area in XPS and AES experiments is now under consideration by the ASTM E-42 Committee.

c. Other Cooperative Projects

A listing is given here of SCAWP projects that have recently been initiated. Formal proposals describing the work are currently being prepared by the designated project leaders.

- Measurement of spatial resolution in Auger-electron spectroscopy (M. Prutton, UK)
- Development of reference materials prepared by ion implantation (W. Gries, FRG)
- Development of polymer reference materials (C. Le Gressus, France)
- Correction methods for backscattering in Auger-electron spectroscopy. (J.-P. Langeron, France)
- Reference data for sputtering rates in oxides (H.J. Grabke, FRG)
- Intercomparison of Auger-electron energy and intensity measurements (M.P. Seah, UK; CEC support)
- Development of a standard data transfer format (M.P. Seah, UK)
- Multi-technique characterization of vacancies in alumina (C. Le Gressus, France)

Proposals on several other topics are also currently being developed.

d. National Projects

A number of national activities have been initiated that could lead to new multilateral projects:

- Tests of quantitative surface analyses of Au-Cu alloys by Auger-electron spectroscopy (Japan)
- Comparative study of different electron energy analyzers (France)
- Evaluation of an algorithm for fitting sputter-depth-profile data and round robin to compare interface widths measured with an NBS reference material (USA)

e. Conferences with SCAWP Co-sponsorship

Two successful one-day workshops on quantitative surface analysis were held in the autumn of 1986, the first at NBS on October 24 and the second at NPL on November 17. Each workshop was attended by about 100 scientists. There was active discussion at both workshops on the needs for surface-analysis standards.

Two additional meetings are planned for 1987 at which VAMAS SCAWP projects and opportunities will be discussed:

- Microbeam Analysis Society, Kona, Hawaii, July 13-17
- Topical Conference on Quantitative Surface Analysis, Monterey, California, October 30-31

SUMMARY

An overview has been given of the recent growth of surface analysis and of the urgent need for standards for the several techniques in common use. While the ASTM Committee E-42 on Surface Analysis and the IUPAC Working Group

on Surface Analysis are both active in standards development, there are still many needs to be met for reference data, reference materials, and reference procedures in surface analysis.

The SCAWP has provided a convenient opportunity for scientists in the VAMAS states to contribute to the development of needed surface analysis standards. VAMAS has proven to be a convenient and useful mechanism for both communication and cooperation.

The formation of the SCAWP has stimulated the initiation of a number of national projects as well.[7] Multinational cooperative projects have been established from some of these national projects and from other proposals. It is expected that these activities will lead to standards useful for surface analysts.

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• TECHNICAL WORKING AREAS •

Technical Working Area 1

WEAR TEST METHODS

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In the past six months the first VAMAS round robin intercomparison has been completed. The principal results were published in **Wear 114** (1987) 109-130. A full report containing information from all 32 laboratories that delivered their reports before March 1987, and additional information, has been distributed to all participants and their national representatives.

The results show that the repeatability and reproducibility of friction and wear measurements is comparable with that of other engineering quantities provided that the tests are performed under well controlled conditions. These include: specification of the geometry (dimensions and surface finish) of the stationary and moving specimen; specification of the wear track dimension; specification of chemical composition, microstructure, hardness, and surface roughness; specification of the atmosphere and relative humidity; specification of the type of motion, load, sliding velocity, temperature, and sliding distance; application of an appropriate cleaning procedure for the specimen surfaces; specification of the tribological quantities to be measured in terms of type (e.g., specimen wear or system wear) and physical dimension (e.g., length, mass, or volume).

A second round robin, scheduled to begin in 1987, is expected to include samples of silicon carbide, silicon nitride, zirconia, alumina, coated and uncoated steel. Particular emphasis will be given to the test procedure in order to decrease the spread among the results of the participating laboratories and to gain insight into the sources of the differences among the wear test results. Special attention will be given to the control of humidity, since the relative humidity has a considerable influence on friction and wear of ceramics.

Technical Working Area 2

SURFACE CHEMICAL ANALYSIS

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Surface chemical analyses are now important to a large variety of advanced materials, both immediately (e.g., semiconductors, polymers, metals, oxides, glasses) after fabrication, and at various times during their service life for process optimization, failure analysis, and quality control. Although surface analysis methods in use are extremely valuable, standards of practice, reference data, and reference materials are needed so that

measurements of known accuracy can be routinely obtained and so that measurements and information can be reliably transferred from one laboratory to another.

The principal objective of this Working Party is to produce by coordinated effort the reference procedures, reference data, and reference materials necessary to establish standards for surface chemical analysis. Three multinational projects were initiated in autumn, 1985, and an additional eight projects were initiated in autumn, 1986:

1. Development of thin oxide films as reference materials
2. Development of calibration data for the energy scales of Auger-electron spectrometers
3. Procedures for quantitative x-ray photoelectron spectroscopy
4. Measurement of spatial resolution in Auger-electron spectroscopy
5. Development of reference materials prepared by ion implantation
6. Development of polymer reference materials
7. Correction methods for backscattering in Auger-electron spectroscopy
8. Reference data for sputtering rates in oxides
9. Intercomparison of Auger-electron energy and intensity measurements
10. Development of a standard data transfer format
11. Multitechnique characterization of vacancies in alumina

Two successful workshops on quantitative surface analysis with this Working Party as a co-sponsor were held in 1986 (one in the USA and the other in the UK). Each workshop had an attendance of about one hundred scientists; there was active discussion of needs for reference data, reference materials, and reference procedures. A summary of the discussion on such needs at the UK workshop will be published.

A meeting of the national representatives to the Working Party was held in London on November 17, 1986. The next such meeting will be on October 23, 1987, following the 1987 European Conference on Applications of Surface and Interface Analysis in Stuttgart.

Technical Working Area 3

CERAMICS

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The ceramic program of VAMAS is presently focused on two topics: (1) determination of environmentally enhanced fracture, led by Dr. S. Freiman of NBS (USA), and (2) the determination of hardness, led by Dr. R. Morrell of NPL (UK). An intercomparison is being conducted in each area. Alumina bars for one round robin have been supplied by France, whereas alumina discs for the other have been supplied by the UK.

For measurements of the time dependence of strength, each laboratory has received about 80 samples: 50 percent are to be broken in the as-received state and the remaining 50 percent are to be broken after having been indented with a Vickers indentor. Three stressing rates are to be used. For hardness, each laboratory receives two samples, which correspond to two alumina grades. The test includes measurement of the size of

indentations that have already been measured by NPL, and hardness measurements. Both exercises are expected to be completed this summer.

The data which will be collected from those exercises will be used to delineate the effective guidelines for standardized tests. The present tests are limited to alumina materials only, and must be extended to non-oxide materials. Japan has agreed to supply silicon-nitride samples for the strength tests next October.

Technical Working Area 4

POLYMER BLENDS

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Polymer alloys and blends (PAB) represent a new, rapidly expanding class of materials. During recent years, the number of commercialized new blends exceeds that of new polymers by a factor of three; and the trend accelerates. The specific feature of PAB is the presence of distinct phases. There are miscible polymer blends, but these are usually impact-modified by addition of an immiscible component. Were the PAB miscible, test methods developed for homopolymers would suffice.

During the first annual meeting of the Technical Working Party on Polymer Blends in 1985, it was decided to focus on the multiphasal nature of PAB. Three tons of a polycarbonate/polyethylene blend (PC/PE) containing 0, 25, 50, 75, and 100 wt% of PC (or PE) were prepared under closely controlled conditions. The two neat polymers serve as reference points; work with the three incompatible blends will provide information on the effect of the second phase on measurements.

Since widely accepted test procedures for PAB do not exist, it was necessary to establish agreement on basic test methods on resins and products. For processability, the flow and thermodynamic properties are of prime interest. For characterization of the performance, there is a need to establish mechanical performance under both static and dynamic loading. The multiphase nature of PAB has led to the study of morphology and interfacial properties. The mechanical testing is well advanced, with results from UK, Canada, France, Italy, and Japan being evaluated. The results indicate significant influence of processing on anisotropy of mechanical behavior for blends; the homopolymer processed under the same conditions are nearly isotropic. Such specificity of PAB response must be incorporated into test procedure and its generality validated on other PAB samples.

Technical Working Area 5

POLYMER COMPOSITES

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The aim of the program is to evaluate the mechanical properties of composite materials by delamination testing and fatigue testing. At this time two round robins are underway.

1. Delamination Testing

In cooperation with ASTM 300202 the VAMAS working group is developing a spec imen and a testing method to determine toughness criteria in mode I (tension) and in mode II (shear). After initial work with monotonic loading the program will expand to cyclic loading.

Points for investigation are:

- Specimen size and initial crack size
- Initiation of crack growth and methods of measuring
- Strain rate
- Means of measuring displacements and loads
- Methods of analysis
- Presentation of results

A unidirectional glass fiber-epoxy composite and an equilibrium woven composite were supplied by Vetrotex in May 1987. The first specimens were sent to the participants in April.

In order to provide comparison data for another material, Toray supplied at the same date, 160 specimens in T 300-epoxy composite. Laboratories from France, Japan, the United Kingdom, and the United States are presently involved in this round robin.

2. Fatigue Testing

A program on fatigue testing is being conducted in order to establish recommendations for specimens and testing methods for fatigue limits of glass fiber and carbon fiber composites.

Several different comparisons are undertaken between laboratories in pure stress state results for tension with flexure test results.

The fatigue test parameters are:

- Applied conditions: frequency, wave type, R ratio
- Specimen design
- Mode of loading: tension and bending
- Load control or stroke control
- Environment

Specimens in unidirectional and equilibrium woven glass fiber-epoxy composite have been supplied by Vetrotex; carbon fiber-epoxy composite specimens are to be furnished by Toray. Laboratories from Canada, France, Japan, and the United Kingdom are participating in this round robin.

The next meeting of the international working group on polymer composites is planned on July 21 at Imperial College.

Technical Working Area 6

SUPERCONDUCTING AND CRYOGENIC STRUCTURAL MATERIALS

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An intercomparison of critical current measurement in niobium (3) tin is underway with samples furnished by Japan, the USA, and the European Communities. Twenty-six public, industrial, and university laboratories in Japan, Europe, and the U.S. are taking part. Preliminary results of the test will be discussed at the next Technical Working Party meeting (Boulder, June 1987). The goal of this test is to establish a consensus on standard methods for measuring superconducting critical current at high magnetic fields. The round robin will be completed this fall.

Discussions on the measurement of superconducting AC losses, the stress effects, and the measurement of critical current exceeding 1000 A are being held. Decisions on experimental programs in these areas will take place at the next meeting of the Technical Working Party.

An intercomparison of the tensile test at 4 K is also underway on SUS316LN and YUS170 stainless steels supplied by Japan. Twenty public, industrial, and university laboratories are taking part.

Discussions on the fracture toughness test and the low cycle fatigue test at 4 K on these alloys as well as low temperature shear-test standards for composites are being held.

Technical Working Area 7

BIOENGINEERING MATERIALS

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A round robin test in the area of materials in contact with hard tissues is to be undertaken among participants in Japan, UK, and Italy. Friction and wear tests by the reciprocating pin-on-disc method are being planned. Synthetic hydroxyapatite samples in disk form have been prepared in quantity by a Japanese company under the supervision of Dr. T. Tateishi from MITI. A toxicological evaluation using colony formation of V79 cells *in vitro* also is being considered. The testing methods proposed are being reviewed by laboratories that will participate in the round robin exercise; agreement is expected shortly.

Technical Working Area 8

HOT SALT CORROSION RESISTANCE

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The overall objective of this project is to develop a level of consensus on hot-salt corrosion testing based on a compatible body of corrosion data obtained from rig-testing experiments in various centres in the USA, Europe, and Japan. There is currently no general agreement on procedures for testing of corrosion resistance of superalloys subjected to environments containing NaCl, sulphur, and other contaminants at temperatures up to 1000 °C, and there is a demand from the user industry for an improvement in the situation. Independent efforts have been made in Europe, the United States, and Japan to develop an increased understanding of the corrosion mechanism in these conditions and to rationalize testing practices, and the VAMAS initiative has provided the opportunity to harmonize these activities and move toward broad agreement on test procedures.

A meeting of the Technical Working Party was held at NPL, Teddington, UK, on 20 January 1987; representatives of all participating groups took part in the discussions. Data from the questionnaires on test conditions that had been sent to users of rig-testing equipment had been assessed and a "standard" set of test conditions could be defined. This information provides the basis for an intercomparison, which is planned to confirm that similar levels of corrosion attack occur in each rig when operated in the specified conditions.

Agreement was reached on the details of the intercomparison covering alloys and coatings to be used, design of testpiece, and analysis techniques to be applied after exposure. Arrangements for preparing and coating the necessary testpieces and plans for distribution were agreed. The aim is to have all the materials ready for distribution by June 1987; at that time a draft procedure will be available and the intercomparison will be carried out under the conditions defined.

Technical Working Area 9

WELD CHARACTERISTICS

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A measurement program has been agreed for the study of the influence of trace elements on variable weld penetration in steels. The aim is to provide a data base for the definition of specification limits for surface active elements. The first phase of the program will consist of a "round robin" intercomparison designed to test the consistency of various current methods of TIG welding in terms of weld penetration in well-characterized steels.

Two batches of both 304 and 316 steel are available from the National Research Institute for Metals, Tokyo, and will be distributed to the various participants for measurements of weld penetration, weld-pool temperatures, and surface tension. A protocol for the measurement program is currently being drawn up by Dr. Nakamura (NRIM) and will be sent to the participants in the USA (5), Japan (5), and the UK (3). The level of commitment shown by the various participants will serve as the criterion for a decision to advance to the other phases of the program.

Technical Working Area 10

MATERIALS DATABANKS

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This program is designed to identify standardization activity related to the computerization of material property data. The Task Group has identified many areas where national and international standards bodies need to take action so that the computer databanks of material property data will be compatible and accessible in an easy to use manner.

The final report of this group will appear as the first VAMAS Report in a Supplement to the next issue of this Bulletin. Among the standards that are recommended for development are those for:

- Reporting the full description of engineering materials, test methods, and data reporting, harmonized on an international scale
- Determining the equivalencies of materials, property tests, and data between different countries
- Capturing published data, especially in tables and graphs
- Creating harmonized compilations of terminology and definitions needed to support data systems allowing access to multiple databanks, whether distributed on-line or by personal computer
- Establishing a data interchange format so that data can be exchanged in a convenient, unambiguous way between materials databanks. The DIF would include components both for contents (related to materials data) and for computer-to-computer communications

In addition, several recommendations were made to ensure that the needs of the materials databank community are integrated into standards work of other areas, such as computer networking, applications software, and user interfaces. While our community will not drive these standards development, our needs must be recognized.

Finally, specific recommendations have been made regarding the role of VAMAS in future work in this area.

Technical Working Area 11

CREEP CRACK GROWTH

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The overall objective of this project is to provide a compatible body of technical information on experimental procedures and data evaluation in creep crack growth as a basis for the development of national and international standards. The VAMAS initiative has provided the opportunity for a collaborative framework within which various national and international programmes on creep crack growth can be brought together in order to harmonize the approach to measurement and interpretation of crack growth rates in creep in advanced engineering applications. The following groups are involved:

USA: ASTM, E24 Task Group

EUROPE: European Group on Fracture, Task Group 1
French national programme
United Kingdom Atomic Energy Authority

Japan: Society for the Promotion of Science, Committee 129
National Research Institute for Metals

Work in each of the groups involved is progressing well and a significant volume of data has been produced. A fruitful meeting to begin the task of coordinating the effort was held on the occasion of an international symposium on fracture mechanics at Knoxville in October 1986. At this meeting it was established that there was a need for an extended "Workshop" style meeting to carry out a joint evaluation of the various aspects of the experimental program. Specific tasks were assigned to the members of the Technical Working Party to prepare the ground for such a meeting, and this work is progressing. Preliminary agreement has been reached to hold the meeting over a period of about three days, in Atlanta in July 1987. It is envisaged that the output from the overall program would be agreement on effective procedures for testing and for evaluation of data and a set of data illustrating the value of optimized test and assessment procedures.

Good progress has been made toward the completion of the program of the individual groups and there is a strong industry-led requirement for the development of agreed procedures on this topic which is important in the design and reliable operation of high technology plant. The coordination meeting in July 1987 is seen as a critical stage in the successful completion of the work.

Technical Working Area 12

EFFICIENT TEST PROCEDURES FOR POLYMER PROPERTIES

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This project is concentrating on two areas:

1. Accelerated durability tests for polymers exposed to conditions of heat, light, and/or humidity
2. Correlation between time, temperature, and stress, etc., to provide reliable acceleration of tests or extrapolation of data

The initial efforts consist of (1) a comparison and evaluation of existing methodologies for accelerated durability tests, and (2) a review of correlation procedures and assessment of their future potential. Planning of the contents of two reports is well advanced, and contributions from VAMAS participants are being discussed. The proposed timescale seeks completion of the reports during 1987, including identification of recommendations for further experimental work (for example, round robin tests, scientific examination of mechanisms).

Technical Working Area 13

LOW CYCLE FATIGUE

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Low cycle fatigue (LCF) testing is essential to characterize materials that in service will occasionally be stressed into the plastic region. Currently there are no standardized specimen designs, test conditions, or failure conditions for LCF testing in spite of the fact that these parameters exercise a strong influence upon the results obtained. The object of the intercomparison is therefore to examine the effects of test variables upon results for the various categories of material normally met in practice. The materials selected are as follows:

ALLOY	CYCLIC DEFORMATION CHARACTERISTICS	TEST TEMPERATURE (°C)
IN718 9Cr/Mo	Strain softening	550 ± 2
AISI 316L	Strain hardening	550 ± 2
Nimonic 101 (IN597)	Strain stable	850 ± 2

Following the elevation of the proposal to category B status at the Tokyo Steering Committee samples of the first three materials were sent to Mr. S. Nashimia of NRIM for distribution in Japan. The fourth materials will follow shortly.

Meanwhile in Europe preliminary data on the AISI 316L is being assessed to establish whether any modifications to the intercomparison guidelines are required.

• VAMAS CALENDAR •

Superconducting and Cryogenic Structural Materials Technical Working Party, Boulder	22-23 June 87
Creep Crack Growth Conference, Atlanta	8-10 July 87
Surface Analysis Symposium, in conjunction with the US Microbeam Analysis Society, Kona, Hawaii	13-17 July 87
Polymer Composite Technical Working Party, London	21 July 87
Surface Chemical Analysis Technical Working Party Meeting in conjuction with the European Conference on Applications of Surface and Interface Analysis, Stuttgart	19-23 October 87
Topical Conference on Quantitative Surface Analysis, Monterey	30-31 October 87

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