

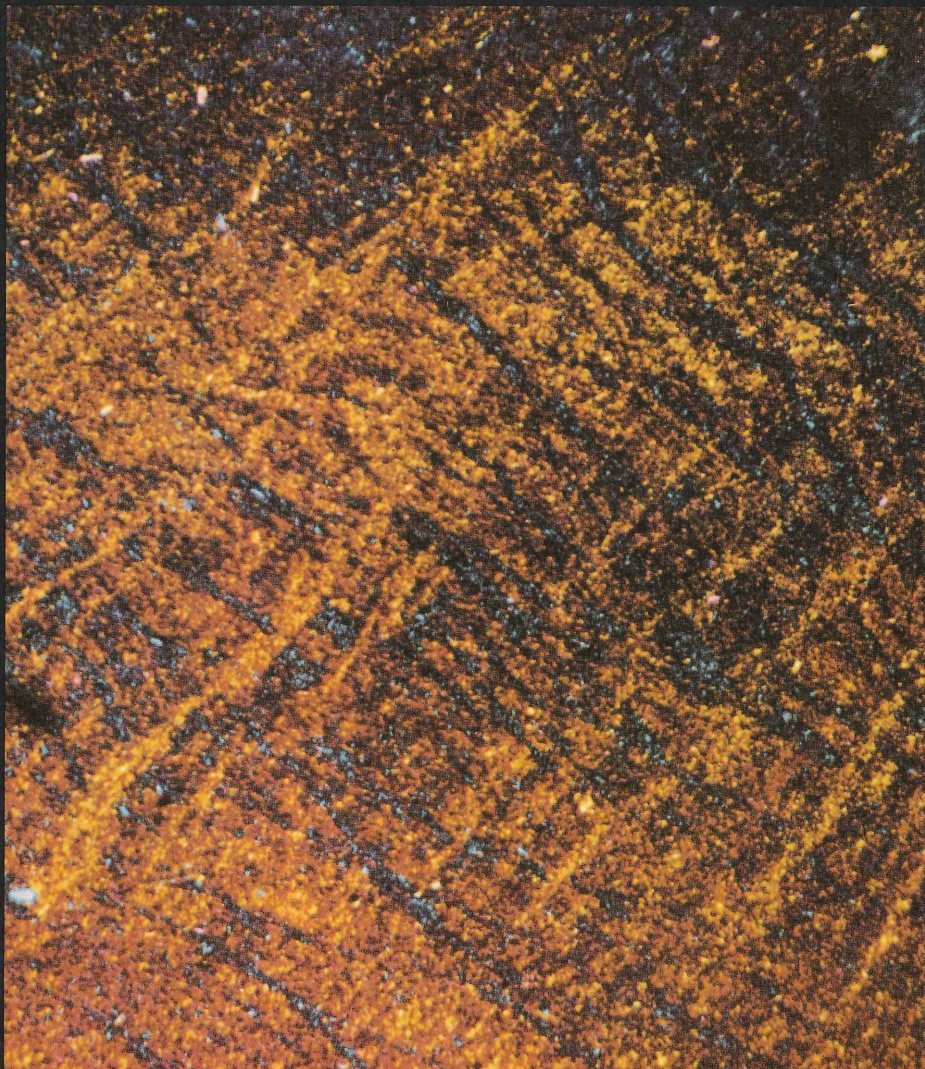
Summit Working Group on Technology, Growth and Employment

• IAMAS •

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

Bulletin No.3

• **January 1986**





VAMAS is a scheme to stimulate the introduction of advanced materials into high technology products and engineering structures with the overall aim of encouraging international trade therein:

- through multilateral research aimed at furnishing the enabling scientific and metrological base necessary to achieve agreement on standards.
- through international agreement on codes of practice and performance standards.

● RECENT DEVELOPMENTS ●

Although the first Working Parties of VAMAS were constituted only two years ago, beneficial effects are already being felt in improved cooperation, and clear patterns of activity are developing.

A central aim of VAMAS is to encourage international collaboration in pre-standards activities but an unexpected consequence has been improved liaison and cooperation within individual countries. This has come about because representatives on Working Parties have consulted industrial, institutional and academic organisations to ensure their needs are adequately catered for and to assess their potential as active participants. As a result there is improved understanding between users and developers of test methods and an enthusiastic support for VAMAS at all levels.

Since the last Bulletin appeared the Steering Committee has met at the Industrial Materials Research Institute near Montreal. Two further Technical Working Parties have been launched, on Creep Crack Growth in High Temperature Alloys and Standards for Computerised Property Data.

Databank standards were envisaged from the start as a potential area for VAMAS. There is a strong emphasis in the proposed programme on the links between the materials and information technology aspects of standards. It is anticipated that one of the consequences of work on this project will be a stimulation of interest in the other Working Parties in standards for presentation of results of tests, so that they can be understood by users, whether human or machine.

One of the most effective ways of determining quantitatively the compatibility of test methods used at different test centres is through the use of round-robin tests in which well characterised samples of materials from the same batch are tested by all participants and the results correlated. Round-robin tests are being used by the Working Parties concerned with Wear, Surface Chemical Analysis, Polymer Blends, Ceramics and Polymer Composites and others are planned.

This will be the last issue of the Bulletin to be produced at NPL for the present. In May 1986, at the next meeting of the Steering Committee in Berlin, the Secretariat will transfer to the National Bureau of Standards. There will be a new format incorporating the VAMAS logo.

● A LOGO FOR VAMAS ●



This attractive new logo has been designed by Carlo Bombardelli, Nora Lanzalaco, and Roberto Merlo, Istituto Europeo di Design of Milan. In the words of the designers the directionality of the mark indicates the convergence of VAMAS towards unification and standards, whilst the intense blue colour and the folio light character set both contribute to a technological image. The intention is to establish the identity of VAMAS with a readily distinguishable symbol, so that its contribution to underpinning the generation of standards and codes of practice for advanced materials is readily recognised.

The logo will come into full use in the next edition of the Bulletin and in the letter head to be introduced when the Secretariat transfers to National Bureau of Standards, USA.

● TECHNICAL WORKING AREAS ●

Eleven Technical Working Areas have been approved. Anyone wishing to learn more about a specific area should contact either the Chairman of the Working Party or his national representative on the Steering Committee.

Technical Working Area

○ WEAR TEST METHODS ○

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At the first Meeting of the VAMAS Working Party, Vancouver, on 19 April 1985, it was agreed to perform a round-robin comparison on the wear behaviour of ceramic alumina and Ni-P coatings together with AISI 52100 steel samples. A test procedure for the wear test was agreed, including specifications of the test system, the materials, the atmospheric conditions and the operating variables in terms of sliding motion, velocity, normal load, temperature and sliding distance.

Altogether, 38 participants from industry, universities and research institutes from all VAMAS countries will contribute to the round-robin programme:

Canada	3	Japan	5
FRG	5	UK	5
France	6	USA	9
Italy	5		

The specimens together with the instructions for the round-robin comparison were sent to the participating institutions in the beginning of August and the first results of the tests are expected to be available in the spring of 1986.

○ SURFACE CHEMICAL ANALYSIS ○

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Surface chemical analyses are made on a large variety of advanced materials (e.g., semiconductors, polymers, metals, oxides, glasses) after fabrication and at various times during their service life for process optimisation, failure analysis, and quality control. Although the 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.

The principal objective of the SURFACE CHEMICAL ANALYSIS Working Party is to produce by coordinated effort the reference procedures, reference data, and reference materials necessary to establish standards for surface chemical analysis. The first meetings of the national representatives to this Working Party took place in Veldhoven, The Netherlands on October 14 and 17, 1985 during the first European Conference on Applications of Surface and Interface Analysis (ECASIA). A number of cooperative projects were discussed and it was agreed that the following should be implemented.

- *Development of thin oxide films as reference materials.*

It is planned to produce in Canada thin films of transition-metal oxides (Fe_2O_3 , NiO , and Cr_2O_3) with thicknesses of about 25 nm on metal substrates. Films of similar thickness of SiO_2 on silicon will be produced in Italy. These films will be characterised by sputter-depth profiling at a number of laboratories in Canada and Italy. The ion sputtering yields of these oxide films under common conditions of sputter-depth profiling will be compared with similar measurements on existing Ta_2O_5 and Ni/Cr reference materials at NPL and NBS.

- *Development of calibration data for the energy scales of Auger-electron spectrometers.*

Current projects exist at both NBS and NPL for the independent measurement of the kinetic energies of the principal Auger-electron transitions of copper, silver, and gold to be used as calibration data in the energy range 50-200 eV. After discussion, the calibration data will be utilised in an interlaboratory comparison of Auger electron measurements to be organised by NPL in 1986.

- *Procedures for quantitative X-ray photoelectron spectroscopy (XPS).*

It is planned to investigate at NBS the suitability of recently fabricated Ta_2O_5 films on silicon and, if necessary, the oxide films prepared above as test materials for an interlaboratory comparison of angle-dependent XPS measurements. If these films can be cleaned adequately with minimal ion sputtering, NBS will organise the comparison of XPS measurements to give information on film thickness and film stoichiometry.

Other projects were discussed and will be initiated following the development of more definite plans. VAMAS has stimulated many national projects and some of these will lead to additional cooperative activities. Finally, two workshops on Quantitative Surface Analysis are planned, one at NBS on October 24, 1986 and the other at NPL on November 17, 1986. Each workshop will precede a major international meeting and will have discussion on four topics: reference data, reference materials, instrument calibration, and analytical methodology. An oral presentation on the scope, organisation, and activities of this Working Party was made to the ECASIA meeting on October 17, 1985.

○ **POLYMER BLENDS** ○

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The Technical Working Party on POLYMER BLENDS has received from General Electric Corp a gift of 1.5 tonnes of polycarbonate. An appropriate grade of linear low density polyethylene was selected on the basis of the flow properties. The resin also has been donated to the Working Party, in this case by ESSO Chemical. In November five compositions containing: 0, 25, 50, 75 and 100 wt per cent of polycarbonate, were prepared in the NRCC Industrial Materials Research Institute. The blends are being formed into two and six mm thick sheets and distributed to Working Party members. Preliminary test results will be discussed during the annual meeting of the Working Party to be held in Montreal, April 7 and 8, 1986. The meeting will follow on the heels of a large international meeting of the Polymer Processing Society. Several members of the Working Party will contribute to this meeting, which includes a symposium on polymer blends.

Proposals for the test procedure to determine melt flow functions, mechanical properties and thermal properties of polymer blends have been received and circulated between members. Those for measurements of morphology, dielectric and dynamic tests are expected to arrive shortly.

○ **CERAMICS** ○

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Time Dependence of Strength and Reliability

A round-robin exercise is under way to determine reliability parameters using constant stressing rate tests. The project will be coordinated by Dr S W Freiman of NBS in close liaison with Dr R Morrell of NPL. About 25 laboratories are participating in the bend tests and 40 indented and 40 non-indented samples will be tested by each laboratory. The alumina samples are being supplied by Pechiney Desmarquest of Montrouge with financial support from the French Ministry of Research and Technology.

Thermal Shock Resistance

This project is in the planning stage. From a theoretical standpoint it appears best to characterise the thermal shock resistance of ceramics through specification of the individual parameters that combine to influence the response to thermal shocks of various types. However, discussions among participants indicate the need to investigate the reliability of established methods for determining the critical temperature drop beyond which impairment of strength results.

Wear, Friction and Hardness

This project is linked with the Working Party on WEAR TEST METHODS and is reported under that heading.

One of the many materials characteristics that go to determine wear characteristics is hardness. A round-robin exercise on hardness measurements is being coordinated by Dr Morrell. Samples indented at NPL will be sent to participants who also will indent them and measure the indentations. The samples will then be returned to NPL for further measurement with the aim of validating the indentation and measurement methods.

Technical Working Area

○ **SUPERCONDUCTING AND CRYOGENIC STRUCTURAL MATERIALS** ○

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At the International Cryogenic Materials Conference held in Boston in August 1985, the Chairman and some of the Working Party discussed the future programme. The first full meeting of the Working Party is scheduled for next April adjacent to the International Cryogenic Engineering Conference in Berlin.

The programme is outlined in VAMAS Bulletin No.2. The cooperative programme will begin with the items on the establishment of standard methods for determining superconducting critical values, in particular those for measuring alternating current losses and strain effects in superconductors, and those for testing the mechanical properties of cryogenic structural materials.

International cooperation in this field is already developing. The Japan-USA cooperative programme on testing of high-field superconductors and cryogenic structural materials as well as similar CEC programme on high-field superconductors have recently been started. These cooperations should be linked and interrelated in the framework of VAMAS.

Technical Working Area

○ **CREEP CRACK GROWTH** ○

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Cracks originating from defects or developing as a result of corrosion or fatigue can grow under steady load conditions in components operating at high temperature. There is a requirement to unify and standardise test procedures for determining creep crack growth rates and for an assessment of correlating parameters (K , C^* etc) to enable data from test-pieces to be applied to complex components. Inter-comparison programmes are in progress in the USA, Japan and Europe and VAMAS provides an opportunity to combine the results obtained. The aim is to collect together the data obtained from the individual programmes and to carry out a joint evaluation of the information. This will enable the relevant test parameters for a standard test procedure to be defined and will provide a compatible technical base for the assessment of correlating parameters. A technical working party has been established and first results should become available during 1986. At that stage procedures for compilation and assessment of the data will be agreed among the various partners.

○ **WELD CHARACTERISTICS** ○

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This program has two components: Weld Toughness and Weld Penetration. Work is continuing on the weld penetration studies as outlined in Bulletin 2. Substantial changes have been made in the weld toughness studies as outlined below.

The weld toughness program has been modified to avoid duplication of effort with a related program of the International Institute of Welding (IIW). Whereas the IIW program is concerned with fracture toughness testing of welds, the revised VAMAS program is concerned with the companion problem of how to specify toughness requirements. The goal is to evaluate various analytical approaches that can be used to calculate required levels of toughness. It is assumed that the IIW and the various national standards bodies will develop appropriate methods for the measurement of toughness. We are considering the next logical question: 'How much toughness is enough?'

The first step has been to request each country to document analytical procedures used to relate fracture toughness requirements to flaw size and stress (strain) levels for the case of welded steel structures. These procedures will be distributed to the participants for evaluation using available data. A meeting is planned in the Summer of 1986 to review the various procedures.

○ **BIOENGINEERING MATERIALS** ○

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The National Representatives of the various countries in the Working Party are: Dr D De Rossi, Chairman, Prof M Josefowicz (France), Dr G Heimke (FRG), Prof P Giusti (Italy), Prof D F Williams (UK) and Dr L Smith (USA). The first organisational meeting of National Representatives took place on 1 June 1985 in Capri immediately before the first meeting of the IUPAC Working Party on 'Interactions of Man Made Polymers with Living Systems' and the 2nd International Symposium on Polymers in Medicine.

The work areas will be classified not by materials type but by the nature of their environment in the body:

- Materials in contact with hard tissues
- Materials in contact with soft tissues
- Materials in contact with blood
- Materials for Biotechnology

A topic of general relevance to be immediately addressed is the establishment of agreed measuring units and definitions in the above areas. Many units at present in use are based on arbitrary definitions not relatable to SI units.

The next meeting of the Working Party will be held in Chester, UK on 3 March, 1986 just before the 'Consensus Conference on Definition of Biomaterials' organised under the auspices of the European Society for Biomaterials.

○ **HOT SALT CORROSION RESISTANCE** ○

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In pursuit of the overall objective of defining an internationally acceptable procedure for assessing the hot salt corrosion resistance of superalloys used in gas turbine applications detailed information on rig operation is being sought from relevant organisations in USA, Europe and Japan. Compilation of this information should be complete in mid-1986 when the next phase of the programme will be initiated. This will involve intercomparison testing of standard materials in prescribed conditions in individual test rigs as a preliminary to specifying test conditions to yield characteristic forms of attack. The overall programme concept has been agreed between partners in USA, Europe and Japan.

○ **POLYMER COMPOSITES** ○

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A programme has been agreed to contribute to the standardisation of prediction of the life-time and delamination of composite materials. The programme will have three components:

- *Survey of test methods*
A survey will be made of existing test methods in tension, compression and bending used for measuring creep rupture, fatigue data and delamination. This survey will be essentially a catalogue rather than an attempt at this stage to provide a critical assessment and comparison. Special note will be made of methods to control and monitor environmental effects.
- *Survey of data*
A general survey would not be appropriate to VAMAS. A more limited survey is required to avoid repetition of earlier work and it will be restricted to materials classes to be covered by the VAMAS round-robin tests.
- *Round-robin tests*
The principal purpose is to obtain a feel for the level of variation that can occur when data on the same materials are obtained by different laboratories using the same or different test methods. The exercise will be restricted to a few parameters. Cooperation with ASTM is beginning on the standardisation of delamination tests.

Material will be provided to participating laboratories who will obtain creep rupture and fatigue S/N curves in uniaxial tension. They will use their existing procedures (including specimen shape and size). It is probable that recommendations will be given on test frequency (10 to 15 Hz), temperature (25°C) and humidity (50%). Final decisions on these and other parameters have not yet been made but will depend on the suggestions received from all countries.

The final choice of materials has also yet to be made. Current proposals are for a commercial fine-weave glass-fibre/epoxy composite (extensively tested at NPL) a corresponding unidirectional material (St Gobain Vetrotex, for example) and CFRP with reinforcement geometry.

Technical Working Area

○ **MATERIAL DATABANKS** ○

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This programme has been started to identify standardisation activity related to the computerisation of materials property data.

The Working Party will concentrate on defining as precisely as possible all standards needed to build materials data systems, whether centralised on mainframes or stored on local or personal computers. The thrust will be to identify problem areas and options for addressing these problems. The group has a twelve-month mandate.

Several specific areas for standards would be investigated, including:

- *Computer Networking:*
What is the present status of the ISO open system interface model?
What other national networking standardisation activities are underway?
What additional work is needed for communication between database management systems?
- *Materials Database Building:*
What standard formats exist for building databases of materials properties?
What groups in which countries have begun addressing these issues?
Do any recommendations or standards exist for the design of materials databases?
- *Materials Data:*
What are the existing standards for materials data reporting?
What materials nomenclature systems exist for alloys, for ceramics, for composites and polymers?
What standards exist for the definition of materials, especially mechanical and corrosion properties?
What activities, formal and informal, are ongoing?
How can harmonisation of terminology be accomplished?

The above list, of course is by no means exclusive. The working group would not only answer these and other questions by identifying what is now happening, but would also identify areas not being addressed.

● TOPIC UNDER CONSIDERATION ●

EFFICIENT TEST PROCEDURES FOR POLYMER PROPERTIES

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The significant dependence of polymer properties on parameters such as time, frequency or rate of loading, temperature, service environment, etc requires provision of appropriate test methods and places a heavy burden on suppliers of materials data. This burden would be eased if more efficient test procedures could be developed, and discussions within VAMAS have focussed on extrapolation of data to longer times and on the development of accelerated tests. The former will utilise the inter-relation between stress, time and temperature for viscoelastic materials, and the latter will be concerned with the durability of materials under environmental exposure. Possible contributions from VAMAS countries are being coordinated with a view to presenting a firm proposal to the Steering Committee for consideration at its meeting in May 1986.

OTHER TOPICS

ECONOMIC CONSEQUENCES OF ADVANCED MATERIALS

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Supported by the French VAMAS team, BIPE has completed a multiclient worldwide project on the economic consequences of new materials (technical plastics, non ferrous new alloys, laminated glass, fine ceramics, special steels...). This study (900 pages) analyses the market, the supply and demand, the strategy and the competition in the world new-material business. The study concludes that in 1983 there was a world market of 80 billion dollars (U.S.A. 35%, Japan 18%, Europe 26%). It will grow at the annual rate of 5.5 percent each year up to 1990. In 1986 BIPE begins a continuous survey of economic development in new materials.

National VAMAS representatives have been asked to indicate their views on the relevance of the study to the VAMAS programme. For further information contact M. Motte.

VAMAS ORGANISATION

VAMAS is co-led by UK and USA. The current Chairman of the Steering Committee is Dr E D Hondros, the alternate Chairman is Dr L H Schwartz, and the Secretary is Dr T I Barry, UK.

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Front Cover:

Optical micrograph of thin section of an extruded 4 mm diameter rod of a 95% alumina ceramic viewed in polarised light through a full wave tint plate. The extrusion axis is horizontal on the micrograph and the banding is due to slippage of the mix on the auger screw. This banding, which indicates orientation of the alumina crystals, is not observable by conventional microscopy, yet it is important in interpreting mechanical and thermal-mechanical property data, emphasising the need for care in the characterisation and specification of ceramic materials. Magnification x50.

Photograph by courtesy of M McNamee of Chloride Silent Power and R Morrell of NPL.