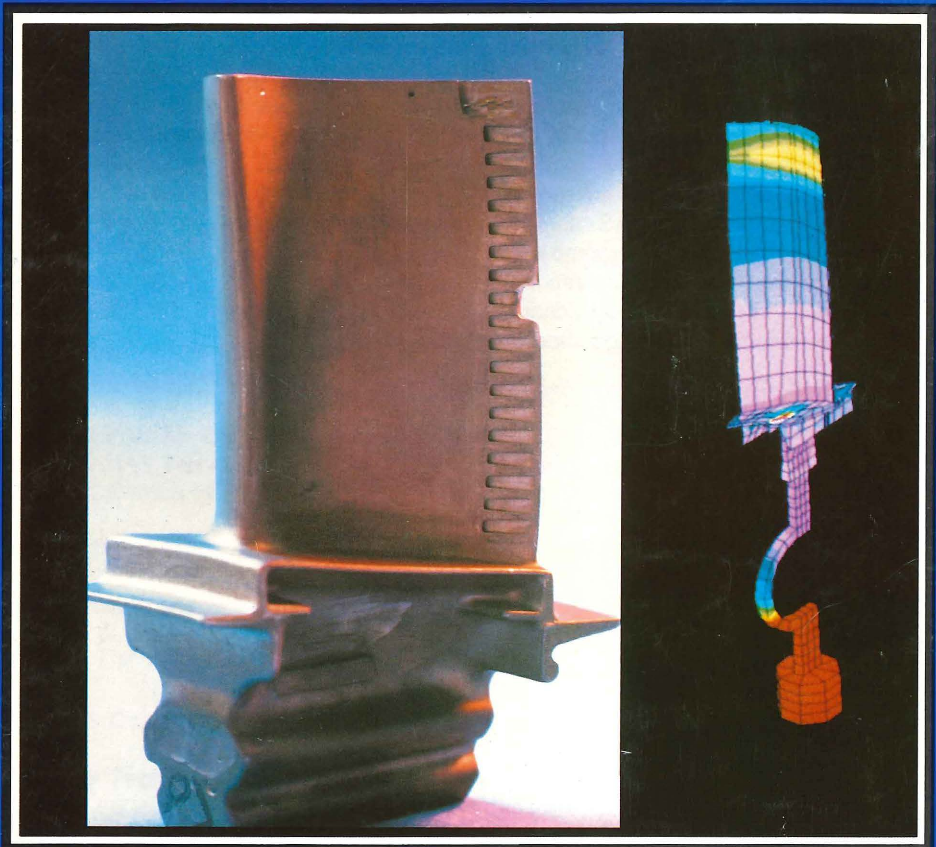
VAMAS



BULLETIN NO. 18
July 1994

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

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: Left - Turbine blade from high performance aircraft engine
Right - Computer simulation of the thermal field during solidification of a single-crystal turbine blade

Photograph courtesy of National Institute of Standards & Technology, Gaithersburg, MD, USA and UES, Inc., Dayton, Ohio, USA.

VAMAS

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• Foreword •

A major focus of this issue of the Bulletin is the summary report on the Joint Steering Committee/TWA Planning Workshop held at the EU Joint Research Centre, Petten, The Netherlands in March 1994. A reading of this summary provides to all VAMAS participants a sense of the breadth of the VAMAS program and the important technological areas whose pre-standardization needs are being addressed, especially those where new TWAs have been initiated.

At the Petten meeting, two new TWAs were established. Several years ago, an advisory group on statistical methods was formed under the leadership of Prof. Gerisch at BAM to provide both advice to TWAs in the design and implementation of round robin studies and to develop/refine appropriate statistical techniques for testing of advanced materials. VAMAS Report No. 13 issued in 1993 is an excellent summary of this early activity. In response to the need to maximize test program efficiency and to optimize allocation of scarce resources, the Steering Committee supported the transformation of the advisory group into a TWA, TWA 18, with a well-defined work program. With the retirement of Prof. Gerisch, Prof. Dr. Klaus Doerffel, Martin Luther University, Merseburg, Germany has been appointed as the new chair of TWA 18 and Mr. Thomas Fritz, BAM, as Co-Chairman. The initial work project will be to develop a generic guide on designing a materials round robin test program.

The second new TWA approved, TWA 19, will focus on the high temperature fracture of brittle materials. Prof. Karl-Heinz Schwalbe, GKSS-Forschungszentrum Geesthacht, Germany was appointed Chair and Prof. Takeo Yokobori, Teikyo University, Japan as Vice-Chairman. The initial work project will investigate crack initiation and growth in intermetallics, titanium alloys, aluminum alloys, and a steel.

In 1991, VAMAS and ISO began to explore the development of a more formal linkage between the two organizations. Recognizing that such a relationship would accelerate the development of international standards on advanced materials, VAMAS and ISO signed a memorandum of understanding in 1993 governing mutual cooperation, particularly with regard to joint publications. A new publication series, *Technology Trends Assessment (TTA),* has been established by ISO to highlight the results of prestandardization research as the basis for new standards. VAMAS has received a significant honor and recognition by having the VAMAS-developed classification system for advanced technical ceramics developed by TWA 14 selected by ISO as the very first TTA document.

Harry L. Rook
Chairman

• Standards Highlights •

VAMAS fosters the development of internationally acceptable standards for advanced materials by existing national, regional, and international standards agencies by providing the technical basis for drafting codes of practice and specifications for advanced materials. A major focus for each Technical Working Area is to further strengthen its ties to the standards-writing community. With the increasing number of concluded pre-standards research projects, it is essential that the results be rapidly transferred to standards-writing organizations. Although not every pre-standards research project produces definitive test results in direct support of a specific standards effort, we continue to see the impact of VAMAS efforts through their recognition in an increasing number of adopted standards.

Standards Highlights identifies draft or adopted standards documents from national, regional, or international standards bodies that are based all or in part on 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.

Recent Adopted standards include:

1] BSI PD 6539:1994, ***Guide to Methods for the Assessment of the Influence of Crack Growth on the Significance of Defects in Components Operating at High Temperature,*** BSI Committee PVE/1. VAMAS contributor - TWA 11, Creep Crack Growth, T. Gibbons, Chairman.

2] ASTM C1286-94, ***Classification for Standard System for Classification of Advanced Ceramics,*** adopted October 1994, ASTM Committee C28, Advanced Ceramics. VAMAS Contributor - TWA 14, Technical Basis for a Unified Classification System for Advanced Ceramics, S. Schneider, Chairman.

Recent Draft standards include:

1] Draft ASTM standard, ***Standard Test Method for the Determination of Fracture Toughness of Advanced Ceramic Materials,*** under development by ASTM Committees C28, Advanced Ceramics, and E8, Fracture and Fatigue. VAMAS contributor - TWA 3, Ceramics, G. Quinn, Chairman.

• Feature Article •

PLANNING FOR THE FUTURE: A REPORT ON THE JOINT STEERING COMMITTEE/TWA PLANNING WORKSHOP

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1 Background

Over ten years ago, the small cadre of individuals who led VAMAS during its birth blazed the trail through their strong belief in the necessity for pre-standards research as a pre-requisite for international standardization and widespread commercialization of advanced materials. This enthusiastic group faced a seemingly infinite choice of barriers to be addressed during the development of materials standards. With the passage of time, the technical leadership within the VAMAS subcommittees or Technical Working Areas (TWAs) changed in response to the evolution of VAMAS as TWAs completed their original goals and disbanded, others identified new challenges and work programs, and new TWAs formed in response to the identification of new standards needs for advanced materials.

In a parallel development, the visibility of VAMAS programs as an important contributor to standards development has grown in part due to the increase in TWA outputs and closer ties to the national and international standards-development organizations, the VAMAS client base. This heightened visibility has re-enforced the importance of technical program planning, evaluation and measurement of program impact at the TWA level to prioritize future directions to efficiently use the voluntary resources available to VAMAS.

To begin the process to develop this global planning focus, the Steering Committee (SC) in 1993 asked the Chairman and Secretary to organize a joint planning workshop with all of the TWA leaders immediately prior to the next SC meeting. The Workshop was held on March 14-15, 1994 at the EU Joint Research Center, Petten, The Netherlands. At this Workshop, each TWA reviewed its near-term and long-term technical program plans and strategies for accomplishing the program goals. The TWA leaders were encouraged to raise issues, concerns, questions, etc. directly to the SC for discussion and resolution. Immediately following the Workshop, the SC convened, reviewed the TWA plans and took action where appropriate.

2 Workshop Structure

The Workshop was organized around oral presentations by each TWA leader to the assembled audience of SC members and TWA leaders. Each presentation was allotted 30 to 50 minutes depending on level of current activity and speakers were encouraged to distribute hard copies of slides or other visual aids used in their presentation. To ensure broad, uniform coverage of topics common to all TWAs, the VAMAS Secretary provided each speaker with an outline of key issues to be covered in the oral presentation. The topics included a review of the current work program and a description of future pre-standardization research needs. The current program review included identification of all current work projects, list of participating organizations, planned completion dates, expected outputs and dissemination strategies, and a critique of terminated, unsuccessful projects. In addition, for current projects with highest priority, technical and non-technical problems were to be discussed. The review of future pre-standardization needs also included identification of the specific need and the planned relationship with national/international standards-writing organizations.

3 Workshop Summary

The Chairman initiated the Workshop with a brief review of the original goals of VAMAS and the evolution of VAMAS over the years into its present configuration. He reiterated the purpose and structure of the Workshop where the speakers were requested to briefly review all current, active work projects, highlight in detail the highest priority projects, describe recent important TWA accomplishments/impacts and how the results can be integrated into the standards development process at the national or international level, and discuss the highest priority new or potential future work projects.

The following discussion represents summaries of the individual oral presentations.

TWA 1 - WEAR TEST METHODS
Dr. E. Santner

The TWA work program contained four active projects. Project No. 1, *Standard Format for Organizing and Reporting Wear Data*, is planned for completion by the end of 1994. Led by Dr. A. W. Ruff, NIST, the project is developing guidelines for data reports to enhance comparability of results from different sources. Laboratories from nine countries are participating in the project. Draft formats and a trial set of data have been distributed for comment. The results will be issued as a VAMAS Report and are intended to be incorporated into standards.

Project No. 2, *Interlaboratory Wear Volume Measurement Comparison*, is planned for completion in the summer of 1994. The project, led by Dr. M. Gee, NPL, is designed to establish measurement precision. Originally, twenty-seven laboratories from nine countries agreed to participate and the project leader plans to initiate the analysis of the data when half of the data have been received. The results are intended for publication in archival journals and are to be used to establish test method precision in standards development.

Project No. 3, *Compilation of Standard Wear Test Methods*, has been completed and the resulting compilation has been released as VAMAS Report No. 14, issued September 1993. Led by Dr. P. Blau, ORNL, over 250 friction and wear test standards, guidelines, and practices recognized by six countries are identified. Individuals from seven countries participated. Although not necessarily complete, the report serves as an informational document, particularly for standards harmonization.

The newest project, Project No. 4, *Interlaboratory Wear Testing of Inorganic Coatings*, is scheduled for completion at the end of 1994. Led by Dr. E. Santner, BAM, the project should establish test method reproducibility for wear testing of coatings. Twenty-eight laboratories from 12 countries are involved; one-third of the participants have completed the tests. The results are expected to be published in archival journals and will be used to establish the need for modifications to testing protocols for coatings.

Future Directions:

The TWA membership has identified six topics for evaluation as potential future work projects, including two tentatively assigned higher priority:

- Statistical techniques for analysis of friction and wear results (high priority)

  Widely recognized problem of significant scatter with tribological data highlights the need for identification of the primary origins as a prerequisite for developing and validating standard test methodologies and analysis protocols. This project presents a clear opportunity for collaboration with the new TWA, TWA 18, Statistical Techniques for Interlaboratory Studies and Related Projects.

- Standard format for organizing and reporting friction and wear results (high priority)

  This effort represents the second phase of Project No. 1, and would be focussed on development of the actual format. Project No. 1, or the first or guideline phase, has illustrated the difficulty in describing a uniform format for the many friction and wear test methods and different material classes.

- Effect of machine dynamics on the results of friction and wear results
- Reproducibility of high temperature friction and wear tests
- Development of reference materials for wear tests
- Validation of abrasive wear test methods; reference materials

TWA 2 - SURFACE CHEMICAL ANALYSIS

Dr. M. Seah

The primary work of the TWA worldwide work program focusses on the three most important and widely-used analytical techniques: Auger electron spectroscopy (AES) - 42%; X-ray photoelectron spectroscopy (XPS) - 33%; and dynamic and static secondary ion mass spectrometry (SIMS) and sputtered neutral mass spectrometry (SNMS) - 25%. The TWA has initiated 31 separate projects directed toward instrument performance, depth scale, spectral interpretation, spectral quantification, and data manipulation. These projects are summarized in VAMAS Bulletins No. 16 [January 1993] and No. 17 [January 1994].

Sixteen Projects, each with from one to nineteen organizations participating, are currently in progress. Thirteen Projects have been completed; work has stopped in two [No. 4 & 7] due to lack of resources. Projects 1 & 27 on development of reference materials are complete. The technical work by the international participants is complete for Projects 2, 9, 10, 22, & 23 and is awaiting acceptance and transfer into standards writing organizations. The technical work by national organizations is complete for Projects 17, 20, 25, & 28; integration at the international level will be carried out in the future. Projects 16 & 31 were completed and the results formed the basis for Project 3.

In 1991, ISO established TC 201, Surface Chemical Analysis, under the chairmanship of the USA and the Secretariat at the JISC. In late 1993, TC 201 formalized its work program and subcommittee structure, previously described in VAMAS Bulletin No.16 [January 1993]. Already, the outputs from Projects 10 and 30 have been submitted to the TC subcommittee on Data Management and Treatment. In addition, the results of Project 2 on calibration of energy scales is under development as an ANSI standard in the USA. Coordination of the TWA's work program with the ISO TC will continue as a result of overlap in membership between the two groups.

Future Directions:

The strength of the current programme is in AES and XPS. Many future directions will naturally evolve in response to the documentary work programme of ISO/TC 201. One very important area of development is that of a standardless, calibration-free dynamic SIMS methodology which, if realized, would lead to a traceable, fully quantitative analytical system that would change instrument design, the need for reference materials, and other aspects of the current protocols. An approach has been suggested already that could be evaluated within VAMAS where well-characterized reference materials are available. In the current situation each analyte in each host needs a separate reference material and, whilst these may be made for the semiconductor industry (Si and GaAs), it is uneconomic in general.

A second future goal is to establish a general methodology for the validation of standard mathematical algorithms. The increasing complexity of software for data analysis and interpretation has increased the difficulty by users in testing software validity. Many variants appear in the technical literature but details of their implementation on the results is not always clear and straightforward.

TWA 3 - CERAMICS
Mr. G. Quinn

Since inception of the TWA, seven major projects have been completed in the areas of dynamic behavior, hardness, room temperature and high temperature fracture, and fractographic and quantitative microscopy. The results of these efforts have been well received by the very active ceramics standardization programs in the USA through ASTM C 28, Advanced Ceramics, in the European Union through CEN TC 184, Advanced Technical Ceramics, and in Japan through the JIS Fine Ceramics Committee. These relationships have positioned VAMAS as an interface between these active national programs and also with the new ISO TC 206, Fine Ceramics.

During the previous 12 months, three round robin test programs have been completed: high temperature fracture toughness; fractography; and fracture toughness by surface crack flexure. The latter two projects moved from inception to completion in about one year.

The *High Temperature Fracture Toughness Round Robin* was organized by the Japanese Fine Ceramic Center in 1990 and completed in 1993. Eight laboratories completed the round robin which tested a number of different specimen geometries at room temperature and 1200 °C. The final VAMAS report, No. 16, was issued in December 1993 and the results widely distributed at meetings in North America and Europe. New technical knowledge as well as development of standards has resulted from this project. One geometry was found to measure fracture toughness independent of testing speed, temperature, or atmosphere and may be suitable for both room and high temperature testing. A Japanese Industrial Standard (JIS) has been prepared based on one of the specimen geometries used in the round robin.

The *Fracture Toughness by the Surface Crack in Flexure (SCF) Method Round Robin* was organized in 1992 by NIST and EMPA in Switzerland to complement a new European Structural Integrity (ESIS) TC 6 round robin in Europe. The testing program was completed by 20 laboratories in September 1993 and a draft of the final VAMAS report, No. 17, prepared. The round robin results revealed that fracture toughness is not strongly sensitive to the precrack size and that new techniques for making and measuring precracks were developed. The consensus of the participants was that the method was more suited for laboratory research measurements rather than as a day-to-day technique.

The *Fractography Round Robin* was organized and completed in 1993 by NIST and U.S. Army Research Laboratory. The overall objectives were twofold: to develop a scheme for finding and characterizing fracture origins in advanced ceramics; and to evaluate the overall effectiveness and applicability of MIL HDBK 790, "Fractography and Characterization of Fracture Origins in Advanced Structural Ceramics". Seventeen laboratories completed the round robin's three topics: Machining damage interpretation by photo analysis; Specimen examination for fracture origin characterization; and Characterization of participant's own materials. A VAMAS final report is currently being prepared. Using preliminary results, a draft fractography standard is under development by ASTM C 28, Advanced Ceramics, with the fractographic definitions already being balloted.

Future Directions:

Taking advantage of its extensive experience with round robin testing projects involving up to twenty four participating laboratories, the TWA has evolved a planning strategy for conducting a successful round robin that may have broad applicability as a planning tool within the VAMAS community. This strategy is based on four fundamental requirements: (a) projects must have well-focussed technical objectives; (b) round robins should be limited to require only one to one and a half staffweeks of effort per participant; (c) reports and data release must be made in a timely fashion; and (d) all participants must have the opportunity to review and revise a draft version of a final report. Requirement (b) is especially important for round robins involving a large number of participants because of the delays in items (c) and (d) that can occur when an insufficient number of participants submit data sets.

The TWA is evaluating the following proposed work projects to establish its immediate priorities:

- a follow-on to the joint CEN/VAMAS Quantitative Microscopy project completed in 1992. Emphasis would be on porosity by point analysis and grain size distribution.
- Surface Roughness to validate a draft Euronorm from CEN TC 184
- Oxidation Resistance with emphasis on mass gain and strength change
- Fracture Toughness of Ceramic Matrix Composites at room temperature using several specimen/test protocols
- Surface Roughness of Ceramic Parts.

TWA 4 - POLYMER BLENDS
Dr. I. Partridge

Initially, the TWA focussed on partially compatible blends of engineering thermoplastics. With the continued evolution of the development of this broad class of materials, a set of generic problems related to standardization have been identified that are relevant to a broader class of materials that, in addition to polymer blends, includes short fiber reinforced thermoplastics and filled polymers. Responding to this technology trend, the TWA has proposed changing its title to Multiphase Polymers as part of its development of an appropriate work program that integrates the narrowly focussed existing program into these new generic topics. The extension of the current work program to the broad class of multiphase polymers raises a number of questions that must be answered. Will one standard mold design be sufficient? Which morphological parameters are critical and need to be measured? Can digital image analysis provide sufficiently unbiased data on such potential measurement parameters as particle or fiber aspect ratio and orientation distribution, and particle or fiber volume fraction or fiber length? Are the mechanical properties strongly dependent on heterogeneity as on anisotropy and what level of heterogeneity is acceptable?

The existing work program has been structured around three aspects of the need to develop a suitable test specimen: morphology quantification (1996 expected completion); specimen mold design (1997 expected completion); and fracture mechanics test protocols (1994 expected completion). [See elsewhere this issue for details] Morphology characterization is important because of the interrelationship between morphology or structure, processing, and properties. These results will be reported through a VAMAS technical report. Quantification of the morphology of polymer materials that form heterogeneous structures through the use of round robin exercises provides guidance for the iterative process of test specimen mold design and the eventual evaluation of one or more molds. The output from this element would lead to the adoption of standard test specimens.

Concurrently, fracture mechanics test development is proceeding through a collaboration with the Task Group on Polymers and Composites of Technical Committee 4 of the European Structural Integrity Society (ESIS). There is a clear need to ensure wide applicability of test methods to commercial engineering polymers. Ultimately, validated fracture test methods are expected to lead to adopted standards through ISO TC 61, Plastics. Since 1990, the VAMAS/ESIS collaboration has led to important progress in four areas: the slow fracture K_{1C}/G_{1C} protocol has been accepted by ISO for further development and the appendix on short fiber reinforced plastics is being revised; the J-integral protocol is in final draft form; the draft impact K_1/G_1 protocol and the draft on essential work on fracture protocol are undergoing review.

Future Directions:

The industrial community has identified the lack of design data on multiphase polymers as one of its critical needs. Present standards for commodity polymers are inadequate for multiphase polymers. New draft ISO standards do not address problems of inhomogeneity and anisotropy. A single specimen source is required for multiphase polymers and thus it is necessary to extend the present ISO proposal on injection molds for small tensile bars and for plates to larger plate sizes. Validated test procedures are needed to quantify the critical morphology elements. The planned redirection of the TWA work program toward multiphase polymers will build on the blends efforts.

TWA 5 - POLYMER COMPOSITES

Dr. G. Sims

The original TWA work program employed round robin testing studies and was organized under three topical areas: (I) *Delamination Crack Growth* (C. Bathias); (II) *Creep* (L. Vincent/K. Kemmochi); and (III) *Fatigue Test Methods* (G. Sims). The delamination crack growth effort focussed on both static loading and cyclic loading.

The delamination crack growth test program was based on ASTM procedures and used both double-cantilevered beam and end notched flexure specimens of combinations of unidirectional and fabric carbon-fiber/epoxy and glass-fiber/epoxy composite materials. A number of individual laboratory reports of the static loading results have previously been issued and the overall program results were summarized in 1990. The analysis of the static data has led to a review of these test methods by ESIS, JIS, and ASTM. Although general agreement was reached on test configuration and test procedure, differences occurred in data interpretation. Follow-on efforts to draft standards have been initiated by ESIS and ASTM. Limited results from the cyclic loading studies have also been issued by individual participants and the final cyclic loading program summary is in preparation. The cyclic results may serve as input to future ASTM standards development activities.

The flexure and tensile creep round robin program on multidirectional carbon-fiber/epoxy composites has been delayed.

The fatigue test round robin program used a number of unidirectional glass-fiber/epoxy and carbon-fiber/epoxy composite materials and carried out assessments of both flexure fatigue and tensile fatigue methods and an intercomparison of the two approaches. Summary reports of the results for the unidirectional oriented materials have been issued. These results have been transmitted to ISO TC 61, Plastics, as a new work item for SC 13/WG 16 on fatigue test methods.

Future Directions:

The TWA believes it can initiate one new project over the coming 12 months and has identified a number of potential new work projects. Potential projects include:

- Advanced Testing Methods for Damage Tolerance based on high reliability and/or damage tolerance evaluation of controlled-damaged specimens
- Composite Machining and Sample Preparation
- Environmental Test Methods to evaluate, for example, the uptake and effect of water, moisture, oils, chemicals
- Thermal Properties, including physical properties and the effect of thermal exposure on both short and long term properties
- Fatigue Testing Under Additional Loading Conditions not previously used
- Through-thickness Properties, including tension, compression, and shear
- NDE Methods such as X-ray, acoustic emission, ultrasonics
- Mixed-mode Fracture Toughness Testing

TWA 7 - BIOENGINEERING MATERIALS

Although neither the Chairman, Dr. T. Tateishi, nor the Co-chairman, Dr. P. Christel, were able to attend the workshop, a brief written summary was provided which outlined the near-term TWA work program.

Future Directions:

The planned work program consists of two parts: (a) wear test of bioengineering materials and (b) cytotoxicity test of wear debris of bioengineering materials. Pin-on-disk wear tests of ultra-high molecular weight polyethylene against yttria partially stabilized zirconia and nitrided Ti-6Al-4V disks will be carried out in pseudo-extracellular fluid. In the second part, the pseudo-extracellular fluid used in the wear test is filtered, inoculated and cultured. Cytotoxicity tests and tests to evaluate Zr ion effects on cell growth will be carried out. Efforts are underway to identify a coordinating laboratory with the necessary capabilities to organize the necessary round robin testing program. The chairman has initiated discussions with Working Group 2 of ISO TC 194 to arrange the inclusion of VAMAS participants in the ISO experimental program.

TWA 8 - HOT SALT CORROSION RESISTANCE

Mr. J. Sillwood for Dr. S. R. J. Saunders

Hot salt corrosion is a common problem for gas turbine engines and in power station boilers. It occurs when metallic materials are exposed to combustion gases that are contaminated with sulfur from the fossil fuel and alkali metal salts from the combustion air. These contaminants combine in the gas phase to form alkali metal sulfates with relatively low melting points that deposit on components at critical temperatures above the melting points but below the dew points of these sulfates. Historically, considerable difficulty has been experienced with developing a test method to assess material performance under these conditions with the result that a number of different test methods were used that produced results that could not be intercompared easily. This VAMAS project, built on these prior efforts, was designed to clarify which critical parameters, contamination flux rate (CFR) or deposition rate, controlled the overall corrosion rate at a given temperature. Burner rig tests were chosen, where fuels are burnt to simulate operating conditions, to determine if the same contaminant flux rate in the laboratory caused similar corrosion rates. A summary of this project is now available as an Interim Final Report.

The round robin test program used both coated and uncoated materials under conditions to produce both Type 1 [900 °C test temperature, broadfront attack, severe internal sulfadation] and Type 2 [700 °C test temperature, pitting attack] corrosion mechanisms. Test materials included two base alloys, IN 738LC and Rene 80, and two coating systems, IN 738LC/ RT22 (platinum aluminide diffusion coating) and Rene 80/ATD2B (Co-21Cr-10Al-0.3Y overlay coating).

Considerably better agreement was observed for burner rigs of different design, when separated into high and low velocity rigs, than in an earlier round robin organized in

1970. However, a number of difficulties were observed, including: the tests are expensive to perform and many of the participants could not carry out the entire test program under the specified test conditions; since the test samples were not exchanged for analysis, the absence of comparison reference samples led to subjective judgements, especially for the coated samples; due to variability in data, some extreme value statistical analysis will be included in the final report; when conducting laboratory tests it is necessary to increase the sulfur levels, particularly for the Type 2 regime, because in a gas turbine the partial pressure of active species such as SO_3 are substantially increased because the operating turbine pressure is high; and the scatter in corrosion rate results at 700 °C was too great to establish any clear conclusions due to insufficient data. Overall, the general conclusion was the establishment of increased validity of the guidelines for burner rig tests. At present CFR provides a good measure of the corrosiveness of the burner rig environment at 900 °C, especially for intercomparisons within high or low velocity rigs.

Future Directions:

As this round robin was the only TWA project, the decision about whether any further work will be planned will await a complete review of the results by all participants.

TWA 10 - MATERIALS DATABANKS
Dr. Y. Monma and Dr. J. Westbrook

The key objective of the TWA has been to assess the role of standards in the flow of computerized materials information. Among the challenges faced by the TWA in executing its pre-standards work program are those generic to other TWAs as well as those unique to computerized databanks. Often the research focus of the project is hard to define in detail because of the different perspectives between the scientific approach of the laboratory and the engineering approach of most data users and can result in delays in reaching agreement on the actual work project. The importance of terminology and definitions becomes critical in the process of exchanging data, especially working with distributed systems. Extensive experience and skills are required for effective and efficient database building. At the same time, the effort is constrained by system obsolescence due to the extremely rapid progress in computer hardware and software technology. Changes such as downsizing hardware from mainframe to PC, availability of distributed systems, LAN-WAN connectivity, Windows, multimedia, and combining databases with knowledge bases through systems integration are occurring simultaneously.

Beginning in 1986, the work program has evolved in three phases. In Phase 1 (1986-1987), the needs and problems were identified through a consensus process and described in VAMAS Technical Report No. 2, *Factual Materials Databanks - The Need for Standards.* Through a series of workshops and publications, numerous approaches to satisfying these needs were proposed and evaluated during Phase 2 (1988-1990). Two major studies were completed, one, *Classification and Designation Systems for Materials* by K. W. Reynard from the UK, and the second, *Interlaboratory Comparison of Computerized Data Evaluation Methods* conducted at NRIM in Japan. In Phase 3

(1991-1994), working toward interoperative materials data systems, a shared task is underway to complete an inventory of Methods/Models for materials data analysis and evaluation. NRIM has responsibility for metals, NPL in the UK is studying polymers, while NIST in the USA has more recently assumed the task for ceramics.

Future Directions:

With the initial three-phase work program drawing to a close, the TWA has assembled a number of options for possible future focus. Prioritization of the next work program should be completed in 1995. Included among the options identified to date are:

- a re-analysis and update of the database needs document, VAMAS Technical Report No. 2 in the light of the rapid changes in user needs and computer systems
- development of standards and codes of practice for use of models/methods of materials behavior based on the ISO-STEP concept
- a joint venture with another TWA to conduct a demonstration with actual data
- how to share materials information through networks [subject of a November 1995 VAMAS Workshop in Tsukuba, Japan]
- need for standard reference materials for validating test machines and data analysis procedures
- the normalization of materials and properties in computerized systems

TWA 12 - EFFICIENT TEST PROCEDURES FOR POLYMERS
Mr. R. P. Brown

Increasingly, materials durability has assumed greater importance when decisions are made related to material selection for new products or material substitution for existing applications. The often prohibitive costs in both time and money of real time, long term tests to directly measure actual lifetime or time to failure has placed strong pressure on the materials community to develop alternative approaches for durability prediction or estimation.

A detailed survey examined the availability, status, and usage in the VAMAS countries of accelerated durability tests with particular focus on testing in aggressive environments such as heat, light, and liquid chemicals. Based on this survey, three interrelated projects were selected: development of a guide to the use of accelerated durability tests (ADT); interlaboratory test program to evaluate standard reference materials (SRMs) for ADT; and guides to the use of standard reference materials in ADT. To leverage the TWA efforts, linkages have been established with a number of relevant standards-writing organizations, including ISO TC 45, Rubber and Rubber Products, and very close ties to ISO TC 61, Plastics, and ASTM G03, Durability of Nonmetallic Materials.

The draft of the *Guide to the Use of Accelerated Durability Tests* was completed in late 1993 and will be issued as a VAMAS Technical Report. The Guide is aimed at assisting the development of an ISO standard. This thorough review includes information on degradation agents and mechanisms, published standards, designing an ADT program, predictive models, variability and uncertainty, heat ageing, weathering, exposure to liquids and gases, ionizing radiation, pitfalls, and simulated design life exposure of products. The TWA is a co-sponsor of the interlaboratory test program to evaluate SRMs in ADT organized by ASTM G03.06, Durability of Pipeline Coatings and Linings with over 20 participating laboratories. The experimental plan was finalized and test pieces distributed. The full analysis of the test results is not yet available. The third project, the development of guides to the use of reference materials, has been postponed pending completion of the round robin.

Future Directions:

At the present time, no future projects are contemplated beyond completion of the three projects related to accelerated durability testing.

TWA 13 - LOW CYCLE FATIGUE
Dr. M. K. Hossain for Dr. D. Gould

High temperature low cycle fatigue is important for the performance of pressure vessels and steam and gas turbines. Since it is often the life-limiting factor, design data requirements are critical and the many test techniques available make intercomparisons difficult. VAMAS joined a European program under the leadership of the now European Union (EU) Community Bureau of Reference (BCR) on the intercomparison of low cycle fatigue (LCF) testing whose objective was to establish a set of high temperature data representative of accepted testing practice, and to eventually identify the important test parameters affecting the repeatability and reproducibility as a basis for developing a standard test procedure.

The test conditions specified included strain ranges, strain rate, fatigue wave shape, temperature, and test piece surface finish. Participating laboratories were free to follow their normal test sequence in terms of test piece form, extensometry, test piece manufacture, test machine and recording equipment. Since material characteristics may have an influence on LCF life, three material classes were chosen and three strain ranges tested for each material: strain-hardening [AISI 316L steel at 550 °C]; strain-softening [either 9CrMo steel or IN718 alloy at 550 °C]; and strain-stable [Nimonic 101 at 850 °C]. Analysis of the interlaboratory test results indicated that repeatability was within a reasonable factor of two while reproducibility was unacceptably large, up to a factor of 60. One important conclusion was the observation that variability in test results was associated with the testing practice rather than materials with variable properties. Independent tests carried out in the UK showed that misalignment/test piece bending was a key factor in data scatter. Errors in temperature measurement/control and strain measurement/control also are believed to contribute to the scatter. The final report, *Evaluation of Low Cycle Fatigue Test Data in the BCR/VAMAS Intercomparison Programme*, has been issued by the EU as EUR 14105EN.

Future Directions:

As originally planned, no further work projects are scheduled for the TWA. The results of the BCR/VAMAS study have led, however, to a proposal to the EU for a very limited amount of additional testing to test the hypothesis of misalignment/bending as a major cause of data scatter. A large number of European laboratories have expressed interest as well as support from Japan if the proposal is accepted by the EU.

TWA 14 - UNIFIED CLASSIFICATION SYSTEM FOR ADVANCED CERAMICS
Mr. S. J. Schneider

The TWA has one project: to identify and assess the challenges to the development of an international classification system for advanced ceramics and to establish a suitable classification structure and mechanisms for system implementation. The approach followed has five elements/goals: to assure early transfer of classification system to private and public sectors; to determine industry's classification practices and preferences; to establish classification framework and scope; construct full range classification system, including machine-readable codes; to transfer the classification system to standards-writing organizations; and publication and dissemination of the full, comprehensive classification system report.

The broad-based character of the TWA membership, including industry, standards organizations, and governments, and its policy of continuously updating all interested parties was designed to smooth the transfer of TWA outputs to users. At the beginning, a worldwide survey of over 300 organizations was taken to identify industry's needs, with the results published in 1990 as a VAMAS report. A workshop was then held to develop the framework for the system development based on the survey results. Among the first tasks was to establish the scope and limits of the intended system, including: a definition of advanced (technical or fine) ceramics; selection of a matrix concept with descriptor fields for the system structure; exclusion of traditional and tonnage ceramics; and inclusion of all product forms of advanced ceramics. Using the services of an outside contractor and financial support from the EU Community Bureau of Reference, the Institute for Standards Research of ASTM, and the Japanese Fine Ceramics Association, the development of a complete classification system was completed in late 1992. The classification system includes coverage of over 1000 product classes, simple classification logic, and a structure that will allow the system to be easily expanded to cover the traditional and tonnage ceramic materials originally omitted from the project scope. Already standards-writing organizations have moved to incorporate the classification system into standards, including an approved ENV standard, an ASTM draft standard, and acceptance of the system by ISO TC 206, Fine Ceramics, as a standards work project. The final report, VAMAS Report No. 15, *Classification of Advanced Technical Ceramics*, was issued in July 1993 and is available to all interested organizations.

Future Directions:

The obvious next step is to broadly test and validate the classification system. This second phase will focus on developing protocols for the testing, demonstration, and promotion of the system. Prototype software will be developed for data input and retrieval and will be used to evaluate the system by creating machine-readable data tables from actual data sets and retrieving specific information. A final report will contain programming guideline recommendations to assist implementation by users and will provide a final version of the demonstration software. The approach, expected to require 2 years, is to show the utility of the system to diverse customers using existing databases from multiple sources. Once validated, the global vision is to expand the system to other ceramic classes [such as the traditional and tonnage ceramics, requiring 1 to 2 years], link the system to other (non-ceramic, 2 years) materials classes, and finally, within 6 to 10 years, achieve the universal classification of advanced and traditional materials within one framework.

TWA 15 - METAL MATRIX COMPOSITES
Dr. S. Johnson

Existing experimental testing procedures for metal matrix composites (MMCs) are not well developed or documented. Analytical material models are not currently available to describe mechanical behavior necessary for component design. During the formation of this TWA in 1992, an initial survey of interested participants revealed a strong consensus that the initial focus project should address testing of discontinuously reinforced aluminum (DRA) matrix composites because of availability of material and widespread worldwide efforts to commercialize the material. The materials selected for the initial phase are whisker reinforced DRA and particulate reinforced DRA.

The whisker reinforced DRA material was received as part of the U.S. Air Force Title III program. Unnotched specimens were machined from plate material at NRIM, Japan, and distributed by NPL, UK. Fourteen organizations representing the UK, USA, Japan, Germany, France, and the European Union have agreed to conduct room temperature tensile tests to measure moduli, yield and ultimate strength, and strain to failure. The preliminary results of these initial tests are expected to be reported by the end of 1994.

To date, particulate reinforced DRA material has not yet been obtained for parallel tests but the same laboratories conducting the whisker material tests are expected to participate when this material is available.

Future Directions:

Following completion of the room temperature tensile tests on the whisker material and the anticipated initiation and completion of the room temperature tests of the particulate reinforced material, the participating organizations plan to complete this initial study by carrying out elevated temperature tensile tests and fatigue tests on these two materials.

During the original survey of TWA members, interest was also expressed in titanium matrix composites but because of the high cost and difficulty in obtaining these materials, a decision on initiating a study was postponed to a future date. More recently, funds have been solicited from Europe, Japan, and the USA to purchase material from one or both of the two companies that have quoted a price for producing plate material. Funding commitments have been received from organizations in the UK, USA, Japan, and Germany and planning is underway to establish a non-bureaucratic mechanism for purchasing the material with funds from many sources. Coordination of this effort with parallel activities in ASTM and other organizations will be essential because of high material costs.

TWA 16 - SUPERCONDUCTING MATERIALS
Dr. H. Wada

This TWA on superconducting materials was established in 1993. The Steering Committee approved the separation of the former TWA 6 on Superconducting and Cryogenic Structural Materials into two separate TWA because of the increased growth in technical activities resulting from the discovery of high temperature superconductivity in oxides. A close liaison had been established with the International Electrotechnical Commission (IEC) TC 90, Superconductivity, to facilitate the entry of VAMAS output into international standards. An in-depth review of the history of TWA 6 and TWA 16, the new superconductivity technical working area appeared in VAMAS Bulletin No. 17, January 1994.

The three work projects initiated by TWA 6 on the evaluation and validation of test methodologies for traditional low temperature superconducting materials are rapidly approaching completion: *Critical Current Measurement on Nb_3Sn; AC Loss Measurement on NbTi; and Critical Field Measurement on NbTi.* The planning of a new work program on the characterization and evaluation of the new oxide superconductors has been initiated.

Two round robin intercomparisons were conducted on measuring the dc critical current in multifilament wires of Nb_3Sn. The first study, launched in 1986 and completed in 1989, identified strain as the primary origin of data scatter. Modified testing procedures for the second intercomparison, 1989 to 1992, reduced the scatter by a factor of almost four. After analysis of the second round robin results, the test protocol was finalized as a draft standard measurement method and accepted in 1993 by Working Group 2 of IEC TC 90 for development as an IEC standard.

Round robin intercomparisons were also directed toward AC loss measurements and critical field measurements in ultrathin filamentary NbTi wire. For the AC loss studies, both AC susceptibility and vibrating sample magnetometer techniques were used. The results of the first round robin, 1987 to 1990, were used to revise the wire geometry. For the second round robin, initiated in 1990, wires with systematic differences in wire diameter were used. When completed in 1994, the results will be considered as the basis for a possible draft standard for the IEC. A single intercomparison on critical field methods began in 1992 and is expected to be completed in 1994.

Future Directions:

The adopted strategy for the new research focus on high temperature superconductors has two elements: (a) interlaboratory comparisons on critical current measurement methods first using common samples and different measurement techniques and then using common samples with a specified measurement method; and (b) various complementary studies carried out simultaneously with the round robin studies on reliable measurement methods for critical temperature and critical field, mapping of multidimensional critical surfaces, physical properties, theory, and terminology. The TWA is organized with coordinators for the three geographic areas represented in VAMAS: Japan - Dr. H. Wada, NRIM; Europe - Dr. H. H. J. ten Kate, University of Twente; and USA - Dr. L. F. Goodrich, NIST.

In Phase 1, initiated in 1993, the three local coordinators provided common oxide superconductor test samples and superconductor simulators for a local round robin intercomparison to the participating laboratories within their geographic area. Upon analysis of the Phase 1 test results and implementation of any necessary protocol revisions, a second local intercomparison or Phase 2, expected to begin in 1994, will be carried out. Following completion in 1995 of the local intercomparisons, a general round robin will be conducted in which all participants will receive common samples and will follow the same test protocol. A final VAMAS report is expected in 1997. The anticipated results are intended to be used to prepare a guideline for a critical current measurement method for oxide superconductors which will be submitted to IEC TC 90 for consideration as the basis for a new standard.

TWA 17 - CRYOGENIC STRUCTURAL MATERIALS
Dr. T. Ogata

This TWA comprises the remaining half of former TWA 6 and is concerned with structural materials for cryogenic applications. The two major objectives are: to develop an understanding of measurement of mechanical properties at 4K; and to establish a unified, generic testing protocol. The approach followed is to use a series of round robin intercomparisons, first, to identify problems and errors associated with existing room temperature standards or proposed procedures, and second, to examine the results of one round robin to adjust the next intercomparison to clarify further any remaining problems and refine test procedures.

The initial focus was on the tensile test and fracture toughness test for alloys. Both the tensile test round robin (1986 to 1988) and the fracture toughness round robin (1986 to 1989) were conducted on two Japanese steels, SUS 316LN and YUS 170. Limited scatter was observed and largely attributed to the method of strain measurement. To validate the strain measurement protocol, a round robin on the use of strain gages at cryogenic temperatures and the interpretation of apparent strain was completed in 1989. A second intercomparison on tensile and fracture toughness testing (1989 to 1992) with specified control mode, strain rate, and load cell and extensometer calibration was conducted on the SUS 316LN steel and a titanium alloy, Ti-5Al-2.5Sn ELI. Overall the intercomparison was a successful international cooperative effort. Specifically, the tensile test results showed less than 2% scatter and no effect of specimen size, strain rate, and laboratory, and the draft JIS or ASTM procedures used exhibited few problems. The fracture toughness tests also exhibited low scatter, less than 6%, but several issues remain to be clarified in a final standard, including effects of machine compliance, specimen sidegrooving, and effect of control mode.

Future Directions:

Two additional materials have been identified for future test method development work projects: tensile and fracture toughness testing of aluminum alloy 2219 and compression and shear testing of a glass fiber-reinforced-polymer composite, G-10. A need for cryogenic composite material standards is based on the lack of satisfactory room temperature methods suitable for cryogenic conditions, a change in property and failure mode at very low temperatures, absence of existing data suitable for engineering design, supplier/manufacturer data not comparable on a national or international basis, and the typical batch-to-batch variations in material.

Specimens of the aluminum alloy have been prepared and distributed to the participating laboratories in Japan, Europe, and the USA. Tensile and fracture toughness testing is underway. An interim report on the round robin results is expected in 1995. Potential follow-on work includes fatigue testing and impact testing.

The composite material round robin will have two stages. In the first stage, block material will be distributed to participants who will prepare their own specimens and carry out their preferred interlaminar shear tests and through-thickness compression tests. Test variables will include: test temperature; time/strain rate; loading direction; and specimen geometry. Interim reports of progress in the first stage intercomparison is anticipated in 1994 and 1995. Following data analysis and test protocol revisions, a second stage intercomparison will be conducted with all participants following the same protocol for the shear and compression tests. A final report is planned for 1997 or 1998.

TWA 18 - STATISTICAL TECHNIQUES FOR ADVANCED MATERIALS INTERLABORATORY STUDIES

Dr. H. Czichos

With the retirement of the former leader of this new TWA, Prof. Gerisch, BAM, a period of transition is underway while new leadership is identified. VAMAS Report No. 13, *Statistical Techniques for Interlaboratory Studies and Related Projects*, highlighted the value of modern statistical methods in the design and implementation of experimental programs. One of the primary functions of this TWA is to provide guidance and advice on the design of round robin testing programs. Any TWA planning a round robin should take advantage of the expertise in this TWA in planning their experimental program to maximize the benefits from the often limited resources available for experiments. Continuity in the strong start by Dr. Gerisch and the TWA members has been continued by BAM while efforts are underway to identify a new chairman.

TWA 19 - HIGH TEMPERATURE FRACTURE OF BRITTLE MATERIALS

Prof. K.-H. Schwalbe

Many advanced materials proposed for critical applications at high temperatures in power plant, aerospace, and petrochemical applications are characterized by relatively poor ductility or creep brittleness under creep conditions. Engineers, being asked to design with materials with a tendency to brittleness, need appropriate data and methodologies to describe crack initiation and growth, to predict component life, and to extrapolate performance at high temperatures. There are currently no standards for test methods to measure critical engineering design data or for guidance on the use of these data in design and component life prediction.

This new TWA was approved at the 1993 Steering Committee meeting held at NIST subject to the execution of a modified TWA formation form and an approved project initiation document. The inaugural meeting was held in October 1993 at GKSS, Geesthacht, with participation by representatives from Germany, Japan, UK, and the USA, the Core Group. Efforts are underway to stimulate greater industrial participation in the TWA. A suggestion has been made to change the last part of the title from *Brittle Materials* to *Brittle Metals.*

Future Directions:

The work program adopted at the Geesthacht meeting will complement existing activities underway in Europe, the United States, and in Japan. The TWA goals are to develop a test method for creep crack growth rate (da/dt) for brittle metals using ASTM E 1457 as the starting point, and to increase the understanding of fracture processes through the mechanics and micromechanisms of material behavior. The Core Group will coordinate all testing and evaluation, and will draft the proposed test method, while the actual testing program will be carried out by local groups in various countries.

Five materials have been selected for the initial study with VAMAS participation: Ti alloy 6242 [UK, Germany]; Al alloy 2519 [UK]; TiAl [Japan, UK] ; γ-TiAl [Germany, USA]; and a C-Mn steel [UK]. The titanium alloy (500 °C test temperature) and the aluminum alloy (150 °C test temperature) are already part of an ASTM round robin. A round robin has also been initiated by JSPS with the Japanese TiAl intermetallic (600 °C and 800 °C test temperatures). In the UK, a BRITE EURAM project has been initiated on a C-Mn steel. An interim report on progress to date is anticipated by 1995 and completion of this initial project is expected by 1998.

• Feature Article •

NEW TECHNICAL WORKING AREA
TWA 17 Cryogenic Structural Materials

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1 Introduction

The development of the superconducting technologies requires higher strength and higher toughness structural materials. Moreover, recently a few very large projects, e.g. ITER (International Tokamak Experimental Reactor), with worldwide industrial participation have been initiated. Manufactures and researchers have developed materials, but to encourage such developments, standard characterization methods must first be established for relevant properties of cryogenic structural materials necessary for superconducting equipment, because of difficulties in evaluation of these properties and differences in test results. Firstly, it is not easy to carry out tests or measurements at liquid helium temperature because liquid helium is expensive, temperature is extremely low, and a vacuum insulated cryostat is required. Secondly, since specific heat and thermal conductivity of metals are extremely low at cryogenic temperatures near 4.2K, specimens temperature rises to an uncertain value as a result of plastic deformation at high strain rate or discontinuous deformation at low strain rate. Therefore, greater attention must be paid to testing conditions.

Composite materials, especially graphite-fiber reinforced plastic (GFRP), are useful materials for low temperature applications. However, each research institute conducts mechanical tests on the material using its own method and various tests are used in different countries. Results for bulk materials are not in agreement, even at room temperature. For example, shear testing at 77 K and 4 K yield ultimate strengths that range from 8 MPa to 160 MPa for similar materials. This wide disparity is clearly unacceptable for designers. It is unclear how the results for different test methods might be related to each other, and it is impossible to specify the appropriate values for design calculations. Thus, the standardization of testing methods for the materials is highly desirable. Round robin test programs on composite materials and aluminum alloys have been discussed and approved by the TWA.

2 Background

In response to the request by the VAMAS Steering Committee, a Technical Working Area (TWA) on superconducting and cryogenic structural materials was established early in 1986 as TWA 6. The cryogenic structural materials subgroup in TWA 6 has conducted a series of interlaboratory comparisons of mechanical properties, including tensile properties and fracture toughness, in order to develop an understanding of mechanical-property measurements at liquid-helium temperature (4.2 K) and establish a unified method. Two round robin tests (RRT) have been carried out to date. The first RRT focussed on identifying problems and errors that might occur in testing and used high strength stainless steel (YUS 170 and SUS 316LN). Some interesting results were obtained, including: the necessity of a basic approach to microstrain measurement at liquid helium temperature; load-cell calibration for tensile tests; and the effect of the testing variables on the fracture toughness test. Based on the results of the first RRT program, the second RRT program specified the testing conditions in accordance with the latest draft of the testing protocols and used SUS 316LN stainless steel and a titanium (Ti) alloy. The main purpose of the second RRT was to refine the protocols for tensile tests and fracture toughness tests and revise the draft document (1988) through clarification of the remaining problems under the improved testing conditions. In the second RRT, the tensile results showed good agreement among the participating laboratories. These results prove that there are few problems in cryogenic tensile testing for this class of materials. The standard deviation of fracture toughness for SUS 316LN was also improved under specified testing conditions. Further study is required on the effect of serration and the testing variables of side-grooving, strain rate, and control mode.

At the 7th TWA meeting at Huntsville, Alabama, future work projects were identified as follows: 1) Al alloys; 2) Composite materials; 3) Fatigue testsing; and 4) Charpy impact testing.

An advanced Al alloy was chosen as a test material for tensile and fracture toughness testing to refine the procedures because the alloy has lower strength and toughness compared to SUS 316LN or the Ti alloy. The testing of composites was proposed at the 5th TWA meeting and a special session was held in July 1989 at ICMC to define the state of the art in this area. A composite material was originally proposed by the Rutherford Appleton Laboratory (RAL) and the National Institute of Standards and Technology (NIST), but the task was delayed due to a lack of a lead laboratory with composites expertise, an insufficient number of qualified laboratories to participate, and the higher priority of establishing metals standards.

3 Objectives

The primary goal of this TWA is the evaluation of mechanical properties of advanced cryogenic structural materials at temperatures down to liquid helium temperature. The TWA objectives are:

(1) Exchange of information on mechanical testing methods of composite materials at cryogenic temperatures

(2) Intercomparison of compression and shear properties of GFRP at cryogenic temperatures
(3) Establishment of reliable testing methods for GFRP
(4) Intercomparison of tensile and fracture toughness properties of an aluminum alloy at cryogenic temperatures
(5) Establishment of reliable testing methods for cryogenic structural alloys, and
(6) Establishment of recommendable testing methods at cryogenic temperatures.

4 Deliverables

Results will be disseminated whenever possible including international conferences, such as International Cryogenic Materials Conferences and International Engineering Conferences as well as through relevant international journals. TWA reports will be published as summaries of activities. The final product, a report or a paper, will provide greater confidence for the practical application of advanced cryogenic structural materials.

5 Generic Implementation Plan and Activities

(1) Set up an international TWA consisting of experts from participant organizations
(2) Hold TWA meetings and exchange information on mechanical testing methods for composite materials at cryogenic temperatures
(3) Distribute materials to each participant for RRT
(4) Evaluate the scatter in mechanical properties of GFRP and the aluminum alloy at cryogenic temperatures
(5) Distribute specimens to each participant for the second RRT, and
(6) Establish a reliable testing method at cryogenic temperatures after the RRT.

This implementation plan was adopted at the 8th TWA meeting held on July 12, 1993 in Albuquerque, New Mexico, USA. In attendance were representatives from Germany, Austria, Italy, USA, and Japan. It was agreed to immediately initiate the round robin test program on the composite material. The round robin tests include compression testing in the through-thickness direction, and shear testing in the interlaminar direction. NRIM distributed test materials in the first RRT. The aluminum alloy specimens are ready for testing and have been distributed. Approximately 10 institutes from Japan, UK, USA, Germany, France, Austria, and Switzerland, are participating in these projects on the aluminum alloy and composite material. For more information on TWA17, Cryogenic Structural Materials, and its developing program, interested parties should contact Dr. Toshio Ogata.

• Recent VAMAS Outputs •

EXTENDED ABSTRACT

VAMAS Technical Report No. 17
"Fracture Toughness of Advanced Ceramics by the
Surface Crack in Flexure (SCF) Method: A VAMAS Round Robin"

by G. Quinn, NIST, Gaithersburg, Maryland, USA
J. Kübler, EMPA, Dübendorf, Switzerland
and R. Gettings, NIST, Gaithersburg, Maryland, USA

There is considerable worldwide interest in standardization and improving test procedures for the determination of fracture toughness of advanced ceramics. This study is the third fracture toughness round robin organized by TWA 3. One earlier project was coordinated by the Japan Fine Ceramic Center (JFCC) and featured the Single-Edge Precracked Beam (SEPB), Indentation Fracture (IF), and Indentation Strength (IS) methods. The second project was also coordinated by JFCC and featured high-temperature testing with Chevron Notch (CNB), Single-Edge V-Notched (SENVB), and SEPB methods.

The present round robin uses a different technique, the surface crack in flexure method (SCF), also known as the controlled surface flaw method. This round robin, jointly organized by the National Institute of Standards and Technology (NIST) and the Swiss Federal Laboratories for Materials Testing and Research (EMPA), commenced in November 1992 and concluded in September 1993. Twenty-four laboratories agreed to participate and twenty completed the tests.

The surface crack method follows conventional practice to measure fracture toughness: a specimen is precracked, the specimen is fractured, the precrack size is measured, and the toughness is computed from a stress intensity formula for a well-defined crack geometry. A hardness machine with a Knoop indenter is used to create a flaw in a common flexure specimen. In brittle materials, the indenter not only forms the impression, but also forms a semicircular or semielliptical crack under the surface. The novel aspect of the method compared to metal testing is that the precrack is very small, of the order of the size of the real flaws in a ceramic, and fractographic techniques are needed to observe and measure the precrack. Flexure stress can be measured quite accurately and precisely, but some care and skill is involved in obtaining and measuring the precrack. The precrack can be modelled by a semicircle or semiellipse for which there is extensive literature and a convergence of solutions for the stress intensity factors.

Fracture toughness was measured on three monolithic advanced ceramics: hot-pressed silicon nitride, hot-isostatic pressed silicon nitride, and yttria-stabilized tetragonal zirconia polycrystal (Y-TZP). These materials exhibit different degrees of difficulty in the application of this test method. Most laboratories had little problem with the hot-pressed silicon nitride and obtained very consistent results. The fracture toughness for 107 specimens was 4.6 ± 0.4 MPa√m (mean standard deviation). Reasonably consistent results were obtained for the hipped silicon nitride. The fracture toughness for 105 specimens was 5.0 ± 0.6 MPa√m. There was fair agreement for the Y-TZP. For those laboratories with valid results, the fracture toughness results were 4.4 ± 0.4 MPa√m.

The calculated toughness depends upon the stress at fracture and the precrack size and shape. Since toughness depends upon the square root of crack size, the uncertainty in the size measurement is diminished in the calculated results. The round robin showed that the method was surprisingly robust with respect to the crack size measurements. However, some fractographic experience or skill is a prerequisite for assurance of correct results. New techniques for enhancing precrack detection were identified. These include tilting the specimen during indentation, illuminating from low angles with optical microscopy, using combinations of low and high magnification photographs, sputter coating at a grazing angle, tilting the specimen in the scanning electron microscope (SEM), using the backscattering mode in the SEM, and using stereo SEM photography. A new procedure for precracking materials that are resistant to Knoop precracking has been devised. A Vickers indenter can be tilted and canted to create an oversized Palmqvist crack.

EXTENDED ABSTRACT

"Structure and Fracture of Multiphase Polymers"
by C. Durand, I. K. Partridge, and M. W. Darlington
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Interim Report

Multiphase polymers cannot be used in high technology fields unless their fracture behavior and properties are well known. It has now become clear that a study of fracture properties of multiphase polymers must be done with a detailed examination of the structure. This study [MSc Thesis, C. Durand] contributes to the three major elements of the work program of VAMAS TWA 4, Multiphase Polymers:

- The development of an international standard for molding a plate test specimen. This work included a study of the structure of a glass fibre reinforced polypropylene plate injection molded by the National Physical Laboratory in the UK.
- Concerning the morphology quantification, the study develops a reflection upon image analysis procedures of blend structure and presents the results of a digital image analysis (DIA) achieved on a polyamide/poly(phenylene-ether) [PA/PPE] blend.
- European Structural Integrity Society (ESIS) fracture methods have been applied to two fibre reinforced moldings and two blends of immiscible polymers. A critical evaluation of the applicability of these methods has been carried out and related to the morphology of the test samples.

In particular, Linear Elastic Fracture Mechanics (LEFM), both slow rate and high rate, were applied to glass fibre reinforced polypropylene (GFPP) and to glass fibre reinforced polyarylamide (GFPA) molded planes. Similarly, J-integral tests were carried out on PA/PP (polyamide/polypropylene) and PA/PPE injection molded plates. The application of slow rate LEFM resulted in non-linearity (GFPP) and "pop-in" (GFPA) in the load-displacement curves. Structure analysis of GFPP plaques revealed a slight change in the fibre orientation pattern within the molding.

For the blends, it was found that PA/PPE specimens fully satisfied the J-integral test validity requirements. However, the PA/PP specimens failed the validity criteria due to a non-uniform crack length. Further, it was shown that scanning electron microscopy in conjunction with digital image analysis enabled a morphology analysis of the immiscible polymer blends.

Technical Working Area 3

CERAMICS

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Objectives

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

The 8th World Ceramic Congress, CIMTEC, was held in June 1994 in Florence, Italy. In the session on *Standards and Markets for Advanced Materials* on June 30, four papers representing different activities in TWA 3 were presented:

1] *Fractography and Characterization of Fracture Origins in Advanced Ceramics,* J. Swab, M. Slavin and G. Quinn

2] *Fracture Toughness of Advanced Ceramics: A New VAMAS Round Robin,* G. Quinn, J. Kübler and R. Gettings

3] *VAMAS Round Robin on Fracture Toughness of Silicon Nitride at High Temperatures,* M. Mizuno and H. Okuda

4] *Round Robin on Grain Size Measurements for Advanced Technical Ceramics,* L. Dortmans, R. Morrell and G. De With

POLYMER BLENDS

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Objectives

To provide the technical basis for drafting standards test procedures for new, high performance polymer alloys and blends in five complementary technical areas: melt flows; dynamic testing; thermal properties; morphology; and mechanical properties

1 Introduction

In the context of activities in TWA 4, multiphase polymers are taken as those polymeric materials that produce heterogeneous structures when molded, the size scale of the heterogeneity being above 1 μm. This category thus represents a very large proportion of industrially important engineering polymers such as short fibre filled thermoplastics, mineral filled thermoplastics, rubber toughened polymers and partially compatibilised polymer-polymer blends.

The engineering community has a requirement for design data on multiphase polymers. The emphasis in presentation of such data is shifting from the traditional 'single point' to 'multi-point' data in order that the testing provides information relevant to and usable in the modern computer-aided design packages. Standards for data presentation are being developed by ISO TC61, Working Group 4. There is a current ISO work plan to provide standard 60 mm x 60 mm plate specimens for testing. However, a need for a larger plate molding has also been identified, from which the minimal sufficient complement of standard test pieces required for design data could be obtained.

The validity of any data on samples from morphologically complex polymeric materials will have to be underpinned by agreed methods of morphology and mechanical property characterizations. At the present moment, no standards exist on morphology characterization in multiphase polymers. Mechanical property measurement methods commonly applied to polymers exhibit varying degrees of reliability when used for multiphase polymers. The greatest sensitivity to the heterogeneous sample structure is observed in fracture tests.

2 Current Projects

2.1 Plate Molding Development

The usual problem in evaluating the structure and properties of multiphase polymeric materials is that apparently small changes in molding conditions can result in very substantial changes in the material. Whilst a full solution to this problem is unlikely to exist, the difficulties of valid property intercomparisons may be reduced by a judicious selection of samples originating from a single, sufficiently large, plate molding. For this reason a project on development of a 'standard' mold for large plate moldings (150 mm x 150 mm, variable thickness in the range 1 mm to 5 mm) has been initiated by the National Physical Laboratory in the UK.

In the first step of the mold optimization, coathanger edge gated 150 mm x 150 mm plaques with nominal cavity thicknesses of 2.65 mm and 4.00 mm have been molded from unfilled and glass fibre filled PP and PBT, at fibre contents of 30 and 20 wt% respectively. The moldings are currently undergoing analysis for structure and levels of mechanical anisotropy.

2.2 Fracture Testing

Activities of the European Structural Integrity Society (ESIS) Technical Committee No.4 over the last few years have led to considerable development of fracture mechanical test methods suitable for relatively homogeneous polymeric materials. These methods include an LEFM test (K_{IC}/G_{IC}) now accepted by ISO. A J-integral test and an Essential Work of Fracture method are being prepared for submission to ISO. Work on extending the application of these protocols to the range of impact speed testing and to multiphase polymers is also proceeding. This last topic is an area of active collaboration between the ESIS Technical Committee and VAMAS TWA 4.

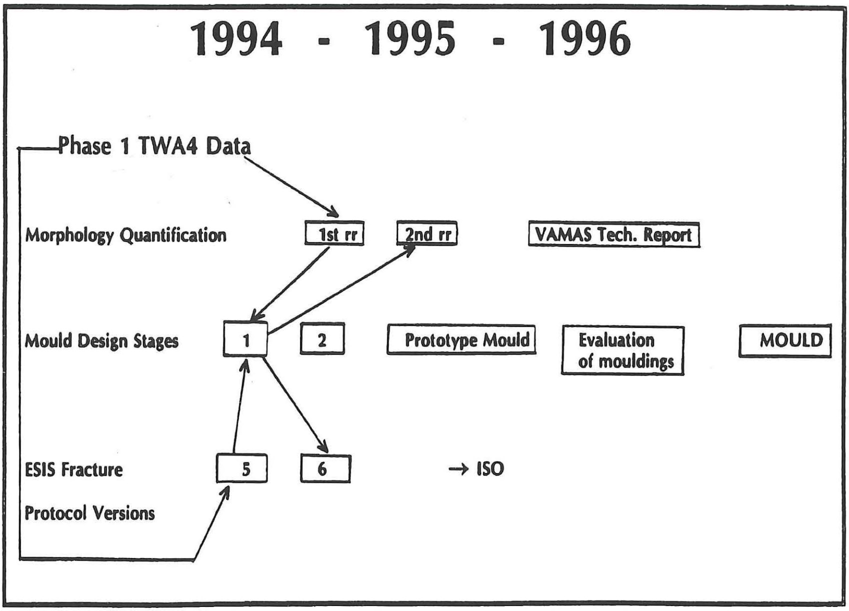
2.3 Morphology Quantification

The ability to describe the morphology of material/specimen in some succinct and reproducible manner is seen as a key pre-requisite to attainment of valid conclusions on the mechanical testing of multiphase polymer moldings and hence on the suitability of individual test specimens and, ultimately, the suitability of test plaque mold design.

In short fibre filled polymers the methods of structure evaluation are reasonably well established. However, the techniques themselves are often very labor-intensive. The rapid developments in Digital Image Analysis (DIA) and in automated image capture afford an opportunity for the future user community to influence the design of commercial software packages at the developmental stages.

A subgroup of VAMAS TWA 4, coordinated from Tokyo University in Japan, employs the techniques of DIA to define methods for quantifying morphology in complex polymer-polymer blends. It remains to be seen to what extent the methodology, developed over many years for the case of the short fibre filled polymers, can be exploited for the even more complicated structures found in moldings made from partially compatibilised blends of engineering polymers.

The figure below is a summary of the plan of action by VAMAS TWA 4 for the near future. It also illustrates the connections between the separate projects outlined above. Enquiries and comments should be addressed to Dr. Ivana Partridge, Chair, TWA 4.



• VAMAS Calendar •

TWA 3 Meeting in conjunction with the 3rd Conference on Fractography of Glases and Ceramics, Alfred University, Alfred, NY, USA	July 9, 1995
TWA 3 Meeting in conjunction with the 6th Symposium on Fracture Mechanics of Ceramics, Nuclear Research Center, Karlsruhe, Germany	July 18-20, 1995
6th European Conference on Applications of Surface and Interface Analysis Montreux Congress Centre, Montreux, Switzerland	October 9-13, 1995

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