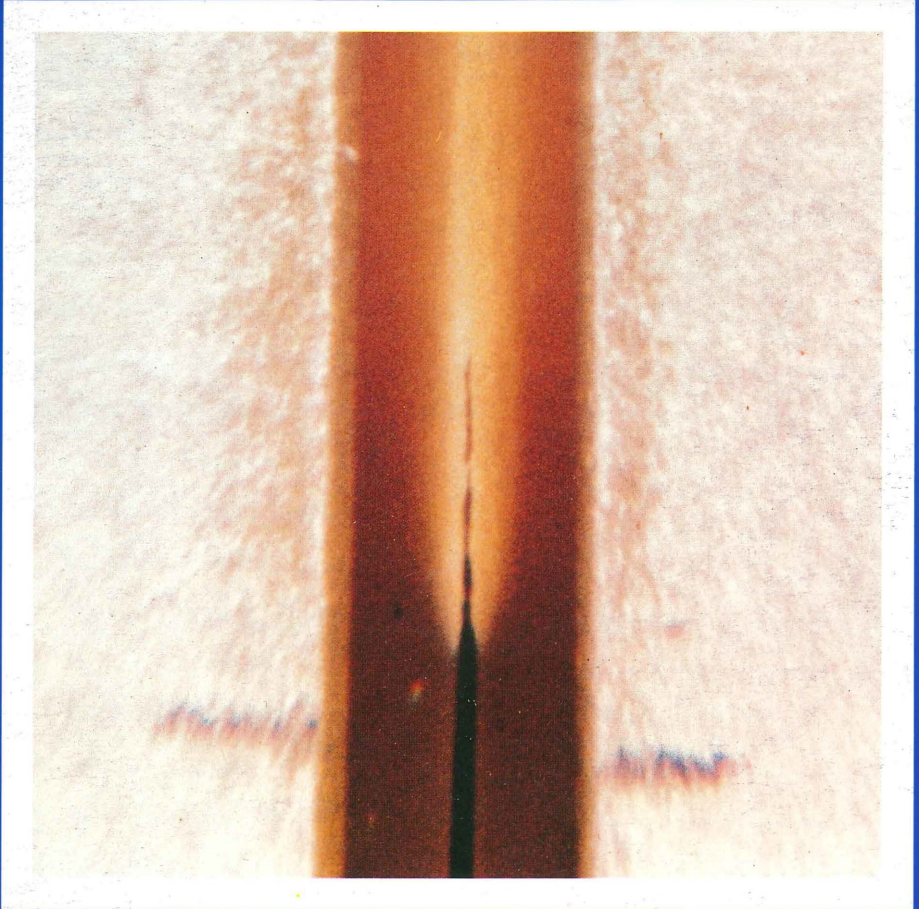




VAMAS



BULLETIN NO. 9

January 1989

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

Cover: Crack tip region in fracture test specimen for composite resin (rubber-toughened epoxy) just before the onset of rapid crack growth. Similar tests on graphite and glass-reinforced composites are discussed in the lead article in this Bulletin. The understanding developed through such testing is essential to the reliable evaluation of tougher advanced composite matrices. *Photograph courtesy of Dr. Donald L. Hunston of the National Institute of Standards and Technology.*



VAMAS

• TABLE OF CONTENTS •

Feature Articles

Recent Results of Polymer Composite Delamination Tests, <i>C. Bathias</i>	1
A New VAMAS Activity on a Unified Classification System for Advanced Ceramics, <i>Samuel J. Schneider</i>	6

VAMAS Technical Note

Thermal Analysis of Polymer Blends.....	8
---	---

Reports of the Technical Working Parties

1 Wear Test Methods, <i>Horst Czichos</i>	9
2 Surface Chemical Analysis, <i>Martin Seah</i>	9
3 Ceramics, <i>P. Boch</i>	13
4 Polymer Blends, <i>L. A. Utracki</i>	14
5 Polymer Composites, <i>C. Bathias</i>	16
6 Superconducting and Cryogenic Structural Materials, <i>K. Tachikawa</i>	17
8 Hot Salt Corrosion, <i>Tom Gibbons</i>	17
9 Weld Characteristics, <i>Tom Gibbons</i>	18
10 Material Databanks, <i>John Rumble and H. Kröckel</i>	18
11 Creep Crack Growth, <i>Tom Gibbons</i>	20
12 Efficient Test Procedures for Polymer Properties, <i>John Lockett</i>	20
13 Low Cycle Fatigue, <i>David Gould</i>	21
14 The Technical Basis for a Unified Classification System for Advanced Ceramics, <i>Samuel J. Schneider</i>	21

VAMAS Calendar	22
-----------------------------	-----------

VAMAS Steering Committee	Inside Back Cover
---------------------------------------	--------------------------

RECENT RESULTS OF POLYMER COMPOSITE DELAMINATION TESTS

Prof. C. Bathias, Université de Technologie de Compiègne
BP 233, F-60206 Compiègne

Two international intercomparisons have been conducted to determine the effect of the initial length of the precrack and the strain rate on the critical strain energy released rate during delamination of composites. The first involved Mode I (opening) delamination of glass fiber composites. The second investigated Mode II (forward shear) delamination of carbon/epoxy and glass/epoxy materials. The tests were conducted on materials supplied by Vetrotex and Toray and tested on Instron machines.

Results from four countries (France, Japan, U.K., and U.S.A.) have been received and summarized in Tables I - IV. All the tests were carried out with DCB specimens described in the VAMAS Program.

Mode I (Opening) Tests

The experiments show that the best representation of the data is provided by a curve that separates initiation of the delamination and its propagation. $G_{1C(0)}$ at initiation is given for the first stage of delamination from the insert. In many cases, delamination growth is characterized by a plateau along which G_{1C} is constant independent of the length of the delamination.

From the various results, we have determined the following values of G_{1C} :

1. Delamination of UD glass fiber composite	$G_{1C(0)}$	300 to 400 N/m
	$G_{1C \text{ plat.}}$	540 to 800 N/m
2. Delamination of woven GFRP	$G_{1C(0)}$	240 to 547 N/m
	$G_{1C \text{ plat.}}$	840 to 1460 N/m
3. Delamination of UD T300 CFRP	$G_{1C(0)}$	80 to 135 N/m
	$G_{1C \text{ plat.}}$	82 to 97 N/m

The scatter of the results is large. Nevertheless useful values of the toughness of composite materials can be derived. The scatter is smaller for CFRP than for GFRP, and ranges between 20% and 100%. The scatter is also smaller for unidirectional laminates than for woven composites.

As was found by ASTM, delamination appears to be controlled by initiation. However, $G_{1C(0)}$ is not always a conservative criterion. For carbon fiber components $G_{1C \text{ plat.}}$ is smaller than $G_{1C(0)}$. Thus, it appears that the most reliable way to study delamination is to use R curve analysis, including the plateau when it is found. For an epoxy matrix, the strain rate has no significant effect on the critical value G_{1C} in the range of 0.1 to 5.

The value of G_{1C} increases a little with the thickness of the woven glass fiber composites and sometimes with the length of the delamination. Therefore it is useful to use a starter with a minimum length of 50mm, at least for unidirectional glass fiber composites.

Mode II (Forward Shear) Tests

For mode II (forward shear) testing it is difficult to separate initiation and propagation, since the delamination is not stable. The average values of G_{11C} that we have determined are:

1. Delamination of UD CFRP	G_{11C} T300	280 to 443 N/m
	G_{11C} T800	571 to 734 N/m
2. Delamination of UD GFRP	G_{11C}	1143 to 2270 N/m
3. Delamination of woven GFRP	G_{11C}	2137 to 2609 N/m

The most important difference between mode II and mode I, between (forward shear) and (opening) delamination, is the instability of delamination in mode II. However, the toughness in mode II is higher than mode I, the amount depending on the materials. The scatter for the mode II tests is of the same order as that for mode I. Specimens precracked in mode I before testing in mode II do not display a clear effect of precracking. More experiments are necessary to clarify this point.

Table 1. Delamination in Mode I for Glass Fiber Composite Materials

Specimen	a mm	V mm/min	G _{1Ca} N/m	G _{1C} mean N/m	Reference
UK Glass Fiber					
51	61.5	0.2		543	M. Maysushima NAL - Japan
52	61.5	0.2		540	
53	61.5	0.2		576	
54	61.5	0.2		661	
55	61.5	0.2		537	
56	61.5	0.2		802	
ave.				610	
UD Glass Fiber					
73	50	0.5	300	650	C. Bathias UTC - France
74	49.5	2	300	650	
76	49.5	5	400	800	
72	35	2	400	800	
71	34.5	2	300		
75	65	2	300	650	
ave.			333	710	
Woven Glass Fiber Thickness 3mm					
E30	50	0.5	365	912	Hunston and Chai NIST - USA
E29	50	0.5	365	912	
E28	50	0.5		912	
E27	50	0.5	240	912	
ave.			323	912	
E58	50	0.2		915	C. Bathias UTC - France
E53	50	0.5		889	
E56	50	1	400	855	
E55	49.5	2		350	
E57	49.5	5		888	
ave.				779	
Woven Glass Fiber Thickness 20mm					
B64	25	0.1	365	1450	Hunston and Chai NIST - USA
B63	37.5	0.1	547	1200	
B61	30	0.5	437	1000	
B60	30	0.5	328	1095	
ave.			430	1189	
B88	19	0.1		841	C. Bathias UTC - France
B89	19	0.2		992	
B87	19	0.5		910	
ave.				914	

Table 2. Delamination in Mode I for Carbon Fiber Composite Materials

Specimen	a mm	V mm/min	G_{ICa} N/m	G_{IC} mean N/m	Reference
UD T300/3601					Hunston and Chai NIST - USA
117-4-1	46	0.5	135	97	
117-4-4	50	0.5	80	91	
117-4-3	46	0.5	134	85	
117-4-2	46	0.5	113	91	
ave.			116	91	
117-6-1	50	0.2		92	C. Bathias UTC - France
117-6-2	47.5	1		90	
117-6-3	61	0.5		84	
117-6-4	34.5	0.5		84	
117-6-5	47.5	0.5		86	
117-6-12	50	0.5		82	
ave.				88	

Table 3. Delamination in Mode II for Carbon Fiber Composite Materials

Specimen	a mm	V mm/min	G_{IIc} mean N/m	Reference
UD Carbon Fiber T800/3631				C. Bathias UTC - France
18113	20	1	734	
18114	20	1	571	
1813 precrack	22	1	775	
18111 precrack	21	1	798	
18112 precrack	25	1	795	
ave.			735	
T300/3601				
187219	20	1	443	
117218 precrack	29	1	280	
117220	29	1	280	
ave.			334	

Table 4. Delamination in Mode II for Glass Fiber Composite Materials

Specimen	a mm	V mm/min	G_{IIc} mean N/m	Reference
UD Glass Fiber				
A51	61.9	2	1850	Y. Noguchi NAL - Japan
A52	61.9	2	1690	
A53	61.0	2	1720	
A54	61.1	2	2270	
A55	61.9	2	1830	
A56	61.5	2	2230	
ave.			1932	
<hr/>				
A84	21	1	1358	C. Bathias UTC - France
A85	21	1	1143	
A86	21	1	1244	
A87	21	1	1226	
ave.			1243	
<hr/>				
Woven Glass Fiber				
E85	21	1	2137	C. Bathias UTC - France
E86	21	1	2530	
E87	21	1	2293	
E88	21	1	2609	
ave.			2392	

A NEW VAMAS ACTIVITY ON A UNIFIED CLASSIFICATION SYSTEM FOR ADVANCED CERAMICS

Samuel J. Schneider
National Institute of Standards and Technology
Gaithersburg, MD 20899 USA

Advanced (fine) ceramics are being extensively researched, developed and brought to market as rapidly as possible. The interest is almost global with significant activity by each country constituting the world's major trading partners. A wide spectrum of standards is, however, lacking and this lack represents one important technical unknown in the commercial market equation. While standards needs are many, foremost is a singular need for a unified classification system built upon standard terminology/nomenclature, as it sets the basis for unanimity in information transfer between researchers, designers, manufacturers, and product users. Further, it sets the framework for product identity and promotion and facilitates the compilation of industry indicators (e.g., sales and production statistics) on an orderly and current basis. Advanced ceramics, being a relatively new industry, with an even newer constituency, has no accepted classification system in place, and what is evolving through common usage is discordant, happenstance and certainly not unified. As a case in point, there is no agreement even for the definition of the term "*advanced ceramics*". The need is worldwide, and without an early agreement on an international classification scheme, conflicting national systems will emerge. The result of independent action will be at best chaotic commercial standards development overall with time lost and product entry to domestic and world markets hampered.

In recognition of this need, the VAMAS Steering Committee at its September 23, 1988, meeting unanimously approved the initiation of a pre-standards activity to support the development of a unified classification system for advanced ceramics. The scope of work provisionally includes the following:

Objective:

To identify and assess the issues inherent in developing a unified classification system for advanced ceramics; establish a building block structure featuring critical elements necessary for international use and establish mechanisms and institutional links as needed and appropriate, between national standards bodies (ASTM, BSI, DIN, AFNOR, CEN, JISC and others) for further system development and refinement to meet individual national and international industrial needs.

Approach:

A working party, representing participating countries and standards groups, will concentrate on three areas: 1. Identification of existing classification schemes (e.g., for metals) for parallel, similar or other materials classes amenable to advanced ceramics, assessing attributes and difficulties; 2. Establishment of a priority hierarchy of development pathways; and 3. Development of a conceptual classification

and terminology critical structure, by adaptation or through new approaches, and determination of the feasibility of a single system for both market indicators and technical elements, considering the following classification factors:

- A.** Organization by end product and use categories (e.g., heat exchanger; structural).
- B.** Categorization by major chemical components (e.g., carbide-SiC/Si).
- C.** Sub-grouping to infer functional attribute (e.g., mechanical).
- D.** Coverage of major product forms (e.g., composite).
- E.** Use of consistent terminology (e.g., toughness).
- F.** Incorporation of property/performance regimes and validation procedures (e.g., mechanical test methods and temperature limits).
- G.** Compatibility with computerized data bases (e.g., computer networking).

Schedule and Outputs:

A two-three year effort is envisioned with the work carried out in serial fashion, roughly divided according to the technical approach elements above:

First 3-6 Months: Startup activities, including organization/confirmation of a working party, initial meeting of members to define issues and develop workplan, establishment of liaison with standards organizations, and initial data (classification) gathering.

Next 6-9 Months: Principal data collection and compilation of results with analysis; preparation for an open/general interest classification workshop to be convened in the summer-fall of 1989 for the purpose of addressing technical approach items #1 and #2 and laying the groundwork for #3. The workshop will cover areas such as: (a) Classification requirements from national/international perspectives; (b) Survey and critique of existing classification systems; (c) Identification of critical classification elements and terminology; (d) Identification (and prioritization) of major advanced ceramic product categories, and (e) Possible skeletal classification scheme(s).

Next 3 Months: Prepare and issue a workshop report for interested parties to summarize workshop findings and to identify next steps.

Next 10 Months: Develop classification system framework, using as guidelines, workshop findings and specific critiques and recommendations from national standards bodies.

Next 2 Months: Prepare and issue final report for individual consideration and action.

VAMAS TECHNICAL NOTE

VAMAS Technical Note #3

THERMAL ANALYSIS OF POLYMER BLENDS

Differential scanning calorimetry/differential thermal analysis (DSC/DTA) measurements have proved to be rapid means to determine the miscibility and the composition of polymer blends. In the case of polycarbonate/linear low density polyethylene (PC/LLDPE) blends, the melting behavior is affected more by the thermal history of the samples than by blending. The glass-transition temperature of PC remained constant, independent of composition, so that the blends were classified as immiscible. The magnitude of these transitions (the enthalpy of melting, and the step in specific heat at the glass transition) were proportional to the LLDPE and the PC content, respectively. The reproducibility and the repeatability of the results from seventeen laboratories were good.

Thermogravimetric analysis (TGA) results on the PC/LLDPE blends showed big differences between the laboratories in the decomposition temperatures, which may be inherent to the different test assemblies used. However, loss of mass up to the end of the different decomposition steps showed minor variations compared with the corresponding temperatures. In the case of PC/LLDPE blends, the decomposition steps are overlapping so that the evaluation of blend composition is complicated. A mutual influence is obvious for the 25% PC-containing blend, for which the PC decomposition cannot be detected. The accuracy for the determination of the blend composition is good when the decomposition temperatures of the blend components differ by more than 50°C.

Conclusions:

1. The experimental conditions for DSC/DTA tests specified in ISO 3146-1985 (E) are suitable for the evaluation of semi-crystalline components of immiscible polymer blends without amendment.
2. The evaluation of the DSC-traces in the region of T_g for amorphous components of immiscible polymer blends needs to be standardized.
3. The experimental conditions for TGA especially the sample preparation and the purge gas flow need to be specified in more detail.
4. The test conditions used for the investigation of PC/LLDPE blends need to be verified for other types of polymer blends.

Seventeen laboratories from six countries contributed measurements of DSC or DTA and of TGA on the blends which had been compounded, extruded, and distributed under the supervision of the National Research Council Canada, Industrial Materials Research Institute. A full publication will be issued in *Polymer Engineering and Science*, mid-September 1988, Vol. 28, No. 17, giving detailed information about the results of each participating laboratory, statistical evaluation, and conclusions on the advantage of thermal analysis for investigating polymer blends.

• TECHNICAL WORKING AREAS •

Technical Working Area 1

WEAR TEST METHODS

Prof. Dr. H. Czichos, BAM, Unter den Eichen 87
D-1000 Berlin 45
Tel: +49 (30) 8104 0020

The second round robin exercise has been nearly completed. The friction and wear behavior of ceramics (silicon-nitride and alumina) and steel were studied. By the end of September 1988 results from about fifty percent of the participating laboratories from all countries included in this intercomparison (Canada, France, Germany, Italy, Japan, United Kingdom, United States of America, Denmark and Finland) had been returned. The evaluation of the results is under way and will be presented for discussion at the conference "Wear of ceramics - Test methods and mechanisms" at the National Physical Laboratory, Teddington, UK, 5-6 December 1988.

The meeting of the National Representatives, which was tentatively scheduled to take place in connection with this conference, had to be postponed following the request from several participants from North America and Japan. The next meeting of National Representatives now is scheduled to take place on the occasion of the international conference "Wear of Materials" in Denver, April 1989.

Technical Working Area 2

SURFACE CHEMICAL ANALYSIS

Dr. M. P. Seah, NPL, Teddington, Middlesex, TW11 0WL
Tel: +44 (1) 943 6634

Progress in this Technical Working Party activity continues strongly, with interest growing as the overall visibility of VAMAS grows. In the previous report we described the interaction of the 18 projects then listed and how they related to the overall requirements of the sector. Those 18 projects are developing, and two further projects are being initiated from Japan. The current list is shown in Table 1. The stage of development of these projects is shown in Fig 1 where, as expected, some projects which have been running for a long time are nearing fruition whilst others are only newly started. It is likely that those reaching fruition will, in turn, spawn new projects.

In previous periods we saw strong progress on narrow fronts, whereas during this period we see a broader steady progress. As projects develop the co-ordinating links shown in Fig 2 become more strongly established. These links were highlighted previously to help project leaders see which interactions would be needed as the projects developed. The links that have been shaded are those that are being or have been properly established to ensure traceability of data from one effort to another.

Table 1. VAMAS SCA PROJECT LIST (1 SEPTEMBER 1988)

Project No:

1. Development of thin oxide films as reference materials (Seah)
2. Development of calibration data for the energy scales of Auger-electron spectrometers (Seah, Powell)
3. Procedures for quantitative x-ray photoelectron spectroscopy. (Powell)
4. Measurement of spatial resolution in AES (Prupton)
5. Development of reference materials prepared by ion implantation (Gries, Gould)
6. Development of polymer reference materials (Le Gressus)
7. Correction methods for backscattering in AES (Landeron)
8. Reference data for sputtering rates in oxides (Grabke)
9. Intercomparison of Auger-electron energy and intensity measurements (Seah)
10. Development of a standard data transfer format (Seah)
11. Multitechnique characterization of vacancies in alumina (Le Gressus)
12. Calibration of surface layers by nuclear reaction analysis (Davies)
13. Tests of algorithms for data processing in AES (Underhill)
14. Tests of algorithms for data processing in XPS (Tougaard)
15. Evaluation of SIMS sensitivity factors (Anderle)
16. EFC Round Robin of Al/Mg oxides (Marcus)
17. Quantitative AES of Au/Cu alloys (Shimizu)
18. Evaluation of LOGIT, an algorithm for fitting sputter-depth-profile data, for the measurement of interface widths of an NIST thin-film reference material (Fine)
19. Round Robin SIMS study of impurities in GaAs crystals (Kurosawa)
20. Round Robin AES study of Pt-Ir alloys (Yoshikawa)

Possible or Proposed Multilateral Activities

Proposed SCA involvement in current CEC projects (Gould)

- A. Reproducibility of ion beam sputtering (Seah)
- B. Granular and planar SiO₂ reference material for XPS (Tran Minh Duc)
- C. Multielement reference material for AES intensity calibration (Seah)
- D. Magnitude and origins of crystallinity effects in AES (Bishop, Le Gressus, Morin, Viehhaus)

Projects Under Development

- E. Tests of peak synthesis and deconvolution algorithms for data processing in XPS (Carley)
- F. Development and test of a procedure to establish on depth scale in SIMS depth profiles (Dowsett)
- G. Reference data for electron attenuation lengths and inelastic mean free paths (Powell)
- H. Development of methods for instrument alignment and calibration in SIMS (Seah)
- I. Databank for Auger-electron and x-ray photoelectron spectra (Powell, Seah)

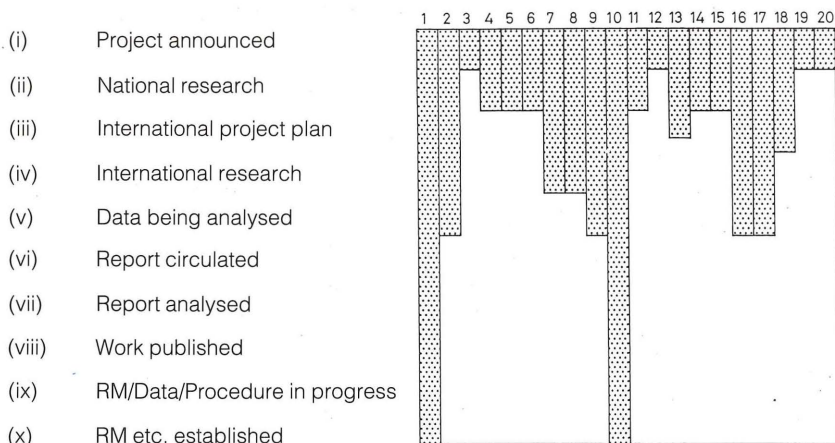


Fig 1. The stage of progress of the SCA individual projects. The length of the bar shows the stage reached.

Project Status

Details of the projects can be found in more detailed reports, which are distributed among participants as follows:

Canada	= 12, 13
France	= 6, 7, 11, 16
FR Germany	= 5, 8
Italy	= 15
Japan	= 17, 19, 20
UK	= 1, 2, 4, 9, 10
USA	= 2, 3, 18
CEC	= 14

International Meetings involving the VAMAS SCA TWP work:

3-7 October 1988: 35th AVS and Topical Conferences, Atlanta Hilton and Marriott Marquis, Atlanta, USA in conjunction with ASTM E-42 Committee Meetings.

15-18 November 1988: Quantitative Surface Analysis, QSA 5, Excelsior Hotel, Heathrow, UK, with TWP Committee Meeting on 14th starting at 6:00 for dinner.

25-29 September 1989: Eleventh International Vacuum Congress and Seventh International Conference on Solid Surfaces, Koln, FRG, in association with the Applied Surface Science Division Sessions.

20-21 October 1989: Topical Conference on Quantitative Surface Analysis at Salem, Massachusetts, prior to 36th AVS meeting 24-27 October 1989, Boston, USA.

24-27 October 1989: ECASIA 89, Palais des Congrès d'Antibes - Juan Les Pins, France.

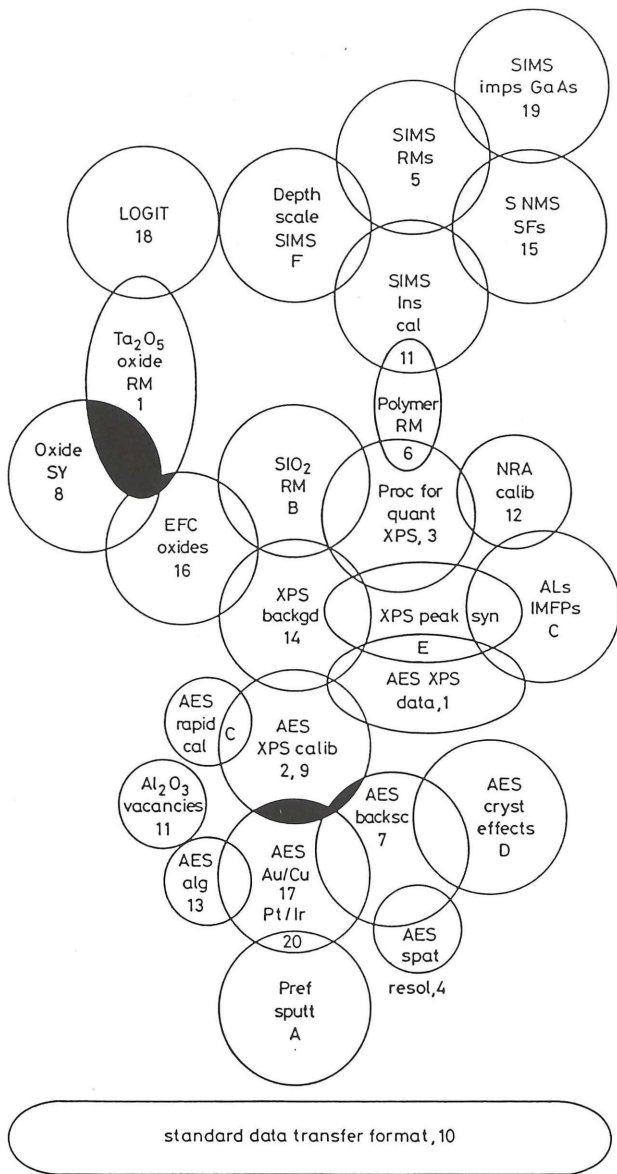


Fig 2. The interaction diagram for SCA projects. The overlaps show the interactions to be established before projects are complete. For simplicity every interaction is not shown. The shaded interactions show some of those where co-ordinating research is in process.

CERAMICS

Prof. P. Boch, ENSCI, 47 av. Albert Thomas, F-87065 Limoges
Tel: +33 (55) 79.34.80

Two interlaboratory studies have now been completed and results are being prepared. The first study was devoted to the characterization of environmental crack growth parameters for alumina samples supplied by Desmarquest-Péchiney. It was organized by S.W. Freiman and E.R. Fuller (NIST). The second study was devoted to the hardness of two grades of alumina ceramics (supplied by British procedures). It was organized by R. Morrell (NPL-UK). A third study on the environmental crack growth parameters for silicon nitride samples supplied by Japan has been initiated.

Two additional areas are under consideration for future studies. The first is thermal shock behavior of ceramics, which is a topic of considerable interest. However, difficulty arises from the fact that studies on thermal shock require special experimental facilities not commonly found in laboratories. Thus, more information must be collected before launching work in this area. At the present time, interest in such a program is found in France, Germany, and Japan.

At a recent meeting of ASTM C28.92 Research Sub-Committee, great interest was shown in a new Japanese technique to determine the value of K_{1C} . Dr. Awaji (Japan Ceramic Center, Nagoya), is now studying the possibility of organizing a VAMAS round robin in this field with Japanese silicon carbide and zirconia samples.

1. "Evaluation of Fracture Toughness for Ceramic Materials by a Single-Edged-Pre-cracked-Beam Method," J. Am. Ceram. Soc., May 1988, pp. 328-333.

Technical Working Area 4
POLYMER BLENDS

Dr. L. A. Utracki, CNR IGM, 75 blvd. de Mortagne
Boucherville, Québec J4B 6Y4
Tel: +1 (514) 641 2280

The 4th annual meeting of VAMAS TWP-PB was held in Orlando, May 12-13, 1988. The first half-day session was devoted to presentation of the test results by the technical coordinators. During the following day and a half, VAMAS members reviewed the last year's progress and planned future activities.

The work on polycarbonate/linear low density polyethylene blends (PC/PE) started in early 1986 after three tons of material was prepared and distributed to nearly 100 laboratories. The results of the last two years activities are contained in 54 individual reports, summarized as follows:

1. Melt Rheology (L. Choplin, Université Laval, coordinating).

Of seven reports, four contained a complete set of numerical data. The steady-state shear viscosity from capillary measurements was found to be strongly affected by morphological changes during the measurements and, as such, unsuitable for characterization of the blends. By contrast, the dynamic-oscillatory tests resulted in excellent repeatability ($\pm 5\%$) and inter-laboratory correlation ($\pm 7\%$). The standardization of melt flow for the purpose of resin characterization should be based on the latter type of testing. The round-robin test results are being prepared for publication.

2. Dynamic Testing of Solids (C. Wippler, Université Louis Pasteur, coordinating).

Eleven reports from four countries were received. There was no practical difficulty associated with the tests. Good agreement was obtained within limits of the experimental accuracy. The blend spectra reflected the transitions in homopolymers. There was a small shift in the dumping maxima that followed no obvious additivity rule. The influence of morphology was reflected in the dynamic-mechanical response; the type of dispersion (drops, rods or lamellae) as well as orientation (parallel or perpendicular to the test direction) could be theoretically calculated. The data can be used as a base for development of a standard test procedure. The round-robin test results have been prepared for publication in *Polymer Engineering and Science*.

3. Thermal Analysis

(G. Pastuska, Bundesanstalt für Materialforschung und -prüfung, coordinating).

An article summarizing the round-robin tests carried out in 17 laboratories from six countries was published in the mid-September issue of *Polymer Engineering and Science*. Excellent repeatability ($T_m \pm 1^\circ\text{C}$ and $T_g \pm 3^\circ\text{C}$) as well as the inter-laboratory reproducibility ($T_m \pm 2.5^\circ\text{C}$ and $T_g \pm 2.8^\circ\text{C}$) were reported.

4. Morphology (E. Butta, Università di Pisa, coordinating).

Ten reports were obtained on optical, scanning and transmission electron microscopy and were in qualitatively good agreement. From these tests the SEM results were most useful. Quantification of the micrographs, especially those showing highly deformed morphology (fibers, lamellae, co-continuous structure) poses serious problems.

5. Mechanical Properties

(I.K. Partridge, Cranfield Institute of Technology, coordinating).

There was good participation in tensile and compression testing. In addition, few laboratories carried out impact resistance and fracture mechanics tests in addition. The tensile testing was carried out in accordance with ISO/DIS 527 or ASTM D638 for homopolymers. The procedure was found applicable for polymer blends. Good inter-laboratory correlation was found with exception of the highest strain rate data. For blends it is important to determine the functional dependence of the mechanical properties over a range of rates and temperatures. For PC/PE only the tensile yield stress slowly varied with strain rate. This relationship could be useful as a tool to classify blends. In the plain strain compression test some of the blends did not produce a distinctive yield point. Several questions remain to be answered, e.g., does the load-displacement curve truly reflect the stress-strain relationship? How do geometry of the sample, strain rate, and the type of material affect the form of the curve? Further work is required. In impact testing, based on the limited data available, the transition from ductile to brittle failure was not apparent. When fracture energy was plotted against the area of new surface, both the British and Canadian laboratories arrived at the same intercept value representing the crack initiation energy at impact, G_{1C} . Cooperation with the European Fracture Mechanics Group was initiated. The round-robin test results are being evaluated for publication.

A second phase of the program is being planned. The aim of Phase II is to provide industry in a shortest time with a set of test methods allowing for quantitative characterization of polymer blends. The methods will be arranged logically as a cascade of tests so that information obtained from one is used in the one to follow.

The work will start by examining the generality of the test methods developed using PC/PE blends in Phase I. For this purpose, several commercial blends will be acquired. Each blend will be selected with respect to its location on the 3 x 3 matrix, consisting of three characteristic criteria: rubbery (R), glassy (G), and crystalline (C), polymer structure. The first blend, offered to the program by ATO Chem, is polypropylene/polyamide (C/C type); the next one will be high impact polystyrene (A/R type) etc. The preliminary program adopted for two years will on one hand lead to a foundation for test standards for melt rheology, dynamic testing of solids, and thermal analysis; and, on another, it will allow progress on those more difficult test procedures such as compressive strength, fracture mechanics, quantification of morphology.

POLYMER COMPOSITES

Prof. C. Bathias, Université de Technologie de Compiègne
BP 233, F-60206 Compiègne
Tel: +33 (44) 20.99.60

The aim of the VAMAS composites program is to evaluate the mechanical properties of composite materials by delamination, fatigue, and creep testing. The results of a recent intercomparison on delamination were summarized in the last VAMAS Bulletin. The program on fatigue testing is now under way and is being led by Dr. I. R. Sced of the National Physical Laboratory. Results from Japan and France are now available. The program on creep testing is being formulated by its leader, Dr. Kemmochi of the Japanese Industrial Products Research Institute.

Delamination Testing

In cooperation with ASTM D30.02.02, the VAMAS working group is evaluating the specimen methodological factors in reliable delamination testing for toughness criterion in opening (mode I) and in forward shear (mode II). The specimens were supplied by Vetrotex and Toray. The program includes glass fiber, carbon fiber, and both unidirectional and equilibrium woven composites.

Results from France, Japan, United Kingdom and USA are presented in the lead article in this Bulletin. For mode I delamination testing in monotonic loading, we concluded that two characteristics determine the delamination resistance. The first is resistance to initiation of delamination (G_{IC0}) from an insert. The second is resistance to delamination growth. The R curve concept has proved most useful in predicting delaminations. For mode II delamination testing, only one factor is relevant, because delamination growth is unstable once initiated. The next step is to study delamination testing under cycle loading.

Other Activity

Programs in fatigue and creep are being formulated and initiated. Those interested in taking part are encouraged to get in touch with the respective leaders or with Professor Bathias.

SUPERCONDUCTING AND CRYOGENIC STRUCTURAL MATERIALS

Dr. K. Tachikawa,
Tokai University, 1117, Kita-kaname, Hiratsuka, Kanagawa 259-12
Guest Researcher of NRIM
Tel: +81 (463) 58 1211

A round robin test of critical current measurements in superconducting Nb₃Sn wires was completed among 24 participant laboratories. The European Community (EC), the United States (US) and Japan each supplied a different type of multifilamentary Nb₃Sn wire. The coefficient of standard deviation for the critical current measurements reported from these labs varied among three samples, and was 6-21% at 12 Tesla. The amount of strain in the specimen at the time of measurement was a major source of scatter in critical current.

A round robin test of AC loss measurement in superconducting Nb-Ti wires was started with 18 participating labs. Four different multifilamentary Nb-Ti wires provided from the EC, the US and Japan were distributed to the participant labs. The measurement will be completed by the end of next March.

In the area of cryogenic structural materials, the round robin test on tensile testing at 4.2 K using SUS 316 LN and YUS 170 steels was finished among 16 participating labs. A round robin test on fracture toughness at 4.2 K using the same steels will be completed by the end of next March. A round robin test on strain gauges measurements at cryogenic temperatures has been proposed in this cooperation area.

The results of the cooperation on superconducting and cryogenic structural materials were reported by the chairman at the recent Steering Committee meeting in Boulder.

HOT SALT CORROSION RESISTANCE

Dr. T. B. Gibbons, NPL, Teddington, Middlesex, TW11 0LW
Tel: +44 (1) 943 6026

The results of surveys of testing practices in the USA and Japan, combined with data available in Europe arising largely from COST50, have been used to draw conclusions with respect to requirements for reliable rig testing of superalloy materials for resistance to hot salt corrosion. This is an important development and represents the first evaluation of the conditions necessary for satisfactory testing of this type. The next phase of the programme will involve an international intercomparison involving all of the participants in the survey from which the conclusions were drawn. The aim will be to evaluate the validity of the conclusions in terms of the uniformity of the results obtained at the various centres; statistical methods will be used to analyze the data. Test materials are being prepared for distribution to the potential contributors. The conclusions may be modified and "fine-tuned" in the light of the results of the intercomparison.

WELD CHARACTERISTICS

Dr. T. B. Gibbons, NPL, Teddington, Middlesex, TW11 0LW
Tel: +44 (1) 943 6026

The programme designed to evaluate the role of trace elements in steels to ensure sound weld penetration in automatic TIG welding has reached the stage where welding experiments have begun. The weld profiles of samples of stainless steels (304 and 316) supplied by the National Research Institute for Metals, Tokyo, with high and low contents of sulphur are being examined at several centres in USA, UK, and Japan. Preliminary data from Japan suggest that the weld profiles are consistent with the view that surface-tension driven flow has a major influence on weld pool fluid dynamics.

A meeting to discuss the data being obtained is planned for London in December 1988.

MATERIALS DATABANKS

Dr. J. Rumble, NIST, Gaithersburg, MD 20899
Tel: +1 (301) 975 2203

Dr. H. Kröckel, CEC Joint Research Centre, ZG-1755 Petten
Tel: +31 (22) 46 5208

VAMAS has been examining the issues related to developing standards for building and using materials databases. VAMAS Working Group 10 has issued (July 1987) a comprehensive report that makes recommendations for action. As a follow-up to these recommendations, three new prestandardization projects have been started by VAMAS in this area, namely:

1. The VAMAS Workshop on Standards for Materials Databanks
2. An Inventory of Materials Designation Systems
3. An Interlaboratory Comparison of Data Evaluation Methods

The VAMAS Workshop on Standards for Materials Databanks

The VAMAS Workshop on Standards for Materials Databanks is being held at the Joint Research Centre in Petten, The Netherlands, on November 15-17, 1988.

The purpose of this workshop is to determine *practicable standardization actions* needed to develop useful and compatible materials databanks and to draw a consensus of needs and priorities to be acted upon by new and existing international and national standards groups. Materials databank builders and networks, national standards organizations, and engineering users in industry and research will all be represented.

The meeting will cover the following topics:

- What refinement is needed for existing materials and materials testing standards because of database building?
- How will needed standards be generated?
- How can harmonization between existing and new standards be accomplished on a national, regional, and international basis?

The means for achieving the above will also be discussed:

- How are national or industrial standards organizations to do this work?
- What is the present activity on standards for materials databanks?
- How can cooperation be fostered on an international basis, e.g., through ISO, CEN, CENELEC, ASTM, or other basis?

The purpose of standards is to make materials databank building easier, to promote compatibility between related databanks built by different groups, and to improve the usefulness to engineering users. Because every industrial country will have public and private groups building materials databanks, standards organizations in these countries will also be concerned with developing standards relevant to their country's interest.

Materials Designation Systems

VAMAS has started a prestandardization project under the leadership of Dr. Keith Reynard of the U.K. to identify, catalog, and describe the major designation systems for engineering materials. This first worldwide inventory of materials designation systems will then be made available to various standards organizations, both national and international, that are developing or reconciling these systems. Contributions to this catalog are welcome, and interested parties should contact the Working Group chairman.

Data Evaluation

A round-robin comparison of data evaluation methods for creep and fatigue data for steel alloys is underway led by S. Nishijima of Japan. The purpose of this international collaborative program is to point out and to quantify the potential problems and discrepancies of "well-accepted" data evaluation methods and the need to establish more standardized methods.

The National Research Institute for Metals has supplied raw materials data, both well-balanced and ill-natured. Properties include creep-rupture and strain-time data and high-cycle fatigue and crack propagation rate data for a sample of welded joints and structural steels. Results will be available in February 1989.

Technical Working Area 11

CREEP CRACK GROWTH

Dr. T. B. Gibbons, NPL, Teddington, Middlesex, TW11 0LW
Tel: +44 (1) 943 6026

In an intercomparison of data, from participants in Japan, USA, and Europe, good agreement on crack growth rates has been obtained with a unified approach to data analysis. The results available are for CrMoV steel and for Alloy 800H, which are important materials for power engineering and chemical plant applications. In addition to data comparisons, the Group has also developed a common perspective on the mechanics of crack growth and has progressed towards a unified approach to calculation of correlating parameters and to experimental methods for crack length measurement.

The current position has been assessed and reviewed in a state-of-the-art report now available in draft form, which should be widely distributed towards the end of 1988. Plans have been laid for a further two-year programme of collaboration in this important area of technology.

Technical Working Area 12

EFFICIENT TEST PROCEDURES FOR POLYMER PROPERTIES

Dr. F. J. Lockett, Consultant,
can be contacted through NPL, Teddington, Middlesex, TW11 0LW
Tel: +44 (1) 943 6024

Following previous discussion and analysis, this project is now concentrating on the problems associated with accelerated durability tests for polymers exposed to conditions of heat, light, and/or water. Details of the initial aspects of the programme were agreed at a meeting in Paris in May 1988, which was attended by representatives of France, Germany, and the UK. Subsequently, Japan has agreed to participate.

The programme is addressing two related aspects: the test procedures themselves and the analysis of data to predict long-term performance from short-term data. In the first aspect, the five participating nations are currently carrying out surveys of testing practices in their countries, identifying the preferred standard or non-standard tests, their principal features, the reasons for their use, the needs for improvement and the potential for harmonisation. These separate national studies will then be compared and combined in a VAMAS report in which conclusions on the need for further work, by VAMAS or elsewhere, will be made.

At the same time, and within the second aspect of the programme, drafts of two reports are being prepared on the status of analytic methods for plastics (by France) and for rubber (by the UK). These drafts will be considered by the full Working Party, and will provide the basis for a VAMAS report identifying the needs for further work. The specific actions described above are intended for completion by the middle of 1989.

As noted in the previous Bulletin, work on the use of the correlation between time, temperature and stress to provide reliable acceleration of mechanical tests or extrapolation of data is not included in the formal VAMAS programme. However, the TWA provides a forum for contact between relevant work in the US and the UK.

Technical Working Area 13
LOW CYCLE FATIGUE

Dr. David Gould, CEC, rue de la Loi 200, B-1049, Bruxelles
Tel: +32 (2) 235 9313

The aim of the program is to assess the influence of test parameters on the performance of alloys that exhibit either strain hardening, softening, or stability during low cycle fatigue testing. On the basis of the results of a round robin, an attempt will be made to define a test procedure that is applicable to the various classes of material.

The work is progressing through an intercomparison involving some eighteen European and ten Japanese laboratories. The European results should be available by the end of this year, with the Japanese ones following in early 1989.

A preliminary assessment of the early European results shows that for both strain softening and hardening materials the design of the specimens affects their lifetimes, with ridged specimens failing significantly earlier than smooth ones.

Technical Working Area 14
**THE TECHNICAL BASIS FOR A UNIFIED CLASSIFICATION
SYSTEM FOR ADVANCED CERAMICS**

Samuel Schneider, NIST, Gaithersburg, MD 20899
Tel: +1 (301) 975 5657

Activity in this area has just been initiated and is described in a lead article at the beginning of this Bulletin.

• VAMAS CALENDAR •

Wear Technical Working Party meeting, in conjunction with International Conference on the "Wear of Materials", Denver	April 1989
Polymer Blend Technical Working Party meeting, Kyoto	April 1989
Steering Committee meeting, Teddington	June 29-30, 1989
Surface Chemical Analysis Technical Working Party meeting, in conjunction with Eleventh International Vacuum Congress and Seventh International Conference on Solid Surfaces, Köln	September 25-29, 1989
Surface Chemical Analysis Technical Working Party meeting, in conjunction with ECASIA 89, Juan Les Pins	October 24-27, 1989
Surface Chemical Analysis Technical Working Party meeting, in conjunction with AVS Meeting, Boston	November 3-7, 1989

• VAMAS ORGANIZATION •

CHAIRMAN (USA)

Dr. Lyle H. Schwartz

Director
Institute for Materials Science
and Engineering
National Institute of Standards
and Technology
Gaithersburg, MD 20899
Tel: +1 (301) 975 5658

SECRETARY (USA)

Dr. Bruce W. Steiner

Institute for Materials Science
and Engineering
National Institute of Standards
and Technology
Gaithersburg, MD 20899
Tel: +1 (301) 975 5655

CANADA

Mr. George Bata

Director
Industrial Materials Research
Institute
75, boulevard de Mortagne
Boucherville, Québec J4B 6Y4
Tel: +1 (514) 641 2280

FRANCE

Prof. Claude Bathias

Université de Technologie de
Compiègne
Département de Génie
Mécanique
Centre de Recherche de
Royallieu
rue P. de Roberval BP 233
F-60206 Compiègne CEDEX
Tel: +33 (44) 20.99.60

M. Pierre Priester

Service "Industries de base"
Association Française de
Normalisation
Tour Europe - CEDEX 7
F-92080 Paris La Défense
Tel: +33 (1) 42 91 57 35

FR GERMANY

Prof. Dr. Horst Czichos

Vizepräsident
Bundesanstalt für
Materialforschung und -prüfung
Unter den Eichen 87
D-1000 Berlin 45
Tel: +49 (30) 8104 0020

Dr. Ing. G. Sievers

Regierungsdirektor
Bundesministerium für
Forschung und Technologie
Heinemannstrasse 2
D-5300 Bonn 2
Tel: +49 (228) 59 555

ITALY

Prof. G. Lanzavecchia

ENEA
Viale Regina Margherita 125
Roma
Tel: +39 (6) 85.28.24.89

Prof. Carlo Rizzuto

Gruppo Nazionale di Struttura
della Materia
c/o Istituto di Scienze Fisiche
Via Dodecaneso 33
Genova 16146
Tel: +39 (10) 5.99.32.45

Prof. Danilo De Rossi

Centro per l'Automatica E Piaggio
Facoltà di Ingegneria
Università di Pisa
Via Diotisalvi 2
56100 Pisa
Tel: +39 (50) 4.44.78/50.08.27

JAPAN

Shizuo Hoshiba

Director
Office of Materials Research
and Development
Research and Development
Bureau
Science and Technology Agency
2-2-1 Kasumigaseki, Chiyoda-ku,
Tokyo 100
Tel: +81 (3) 581 3879

JAPAN (cont'd)

Mr. Yasuhiro Kato

Director
Materials Standards Division
Standards Department
Agency for Industrial Science
and Technology, MITI
1-3-1 Kasumigaseki, Chiyoda-ku
Tokyo 100
Tel: +81 (3) 501 5668

Dr. Kazuyoshi Nii

Deputy Director-General
National Research Institute
for Metals
3-12, 2-Chome, Nakameguro,
Meguro-ku
Tokyo 153
Tel: +81 (3) 719 2271

UK

Dr. Kamal Hossain

Division of Materials Applications
National Physical Laboratory
Teddington
Middlesex TW11 0LW
Tel: +44 (1) 943 6024

Dr. T. B. Gibbons

Division of Materials Applications
National Physical Laboratory
Teddington
Middlesex TW11 0LW
Tel: +44 (1) 943 6026

CEC

Dr. Ernest D. Hondros, FRS

Director
CEC Joint Research Centre,
Petten
Postbus 2
NL-1755 ZG Petten
Tel: +31 (22) 246 6024

Dr. A Garcia-Arroyo

Director DG XII C
Commission of the European
Communities
Directorate General XII
rue de la Loi 200
B-1049, Bruxelles
Tel: +32 (2) 235 11 11

