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



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 emphasises collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardisation 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: A schematic diagram of the bridge compression jig used for pre-cracking ceramic fracture toughness specimens discussed in the feature article by H Awaji and H Okuda



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Foreword •

VAMAS operates under a Memorandum of Understanding signed by senior representatives of government in the seven countries of the Economic Summits and of the Commission of the European Communities. The participating countries, Canada, France, Germany, Italy, Japan, the UK, the USA, and the Commission of European Communities are each represented on its Steering Committee.

Many of our readers will know that the Memorandum of Understanding under which VAMAS operates comes to an end in 1992. Since its inception, nearly 500 individuals from industry, government and academia in the seven summit nations have participated in about 60 projects. This enthusiastic participation on a large scale has demonstrated the clear need for pre-normative research in advanced materials. Success in the technical work and the growing contribution to standardisation have shown the value of the cooperative approach; the level of cooperation and the wider benefits accrued from VAMAS activities have indeed helped VAMAS to gain a unique position in pre-standardisation research.

This has led many of us to believe that we should build on the strengths and achievements of VAMAS. Accordingly, the Steering Committee is currently examining the potential for continuation of VAMAS beyond 1992. In doing so we are looking at the selection,organisation and operation of the Technical Working Areas (TWAs), method of project development, publications, mechanisms for transfer of VAMAS results to standards, relationship with standards organisations and so on. Some of you will have already expressed your views on these issues to Steering Committee members in your own country. It does seem, however, that this is an opportune time for seeking as wide a view as possible from the readers of the Bulletin and all those who have an interest in VAMAS.

The Secretariat would therefore welcome any views or suggestions you may have for future operation and work of VAMAS.

Kamal Hossain Chairman

Feature Article

RESULTS OF THE FRACTURE TOUGHNESS ROUND ROBIN TEST (RRT)

A VAMAS COOPERATIVE PROJECT by H AWAJI, and H OKUDA Japan Fine Ceramics Center Atuta-ku, Nagoya

Introduction

Structural ceramics have excellent mechanical properties, such as high strength, even at elevated temperatures, high hardness, and corrosion resistance. Despite these advantages, the strength of ceramics is extremely sensitive to microscopic defects because of their low toughness. Also, there are some obstacles in evaluating fracture toughness of ceramics such as difficulties in making a precrack, R-curve behaviour, and slow crack growth.

The purpose of the '89 Fracture Toughness RRT in the ceramics working area was to assess methods of measuring fracture toughness parameters of advanced ceramics. The following three methods were examined: Single Edge Precracked Beam (SEPB), Indentation Fracture (IF) and Indentation Strength (IS).

The SEPB method has several advantages, such as theoretical simplicity and good reproducibility for common structural ceramics. This technique uses a "pop-in" precrack arising from a Vickers indent or a straight-through notch. However, the disadvantages are that the technique is useless if a crack front is not visible, and that it is difficult to induce a precrack in some ceramics.

The IF method is known as the most convenient technique. It only needs a small area, and the procedure is quite simple; measurements of the diagonals of a Vickers impression and the accompanying radial crack lengths only are necessary for evaluating the fracture toughness. However, this technique has also several disadvantages such as difficulty of crack length measurement in some ceramics, and the measured fracture toughness apparently depends on the indentation load.

The values of fracture toughness for two materials measured by each participant using three methods were compiled and analysed. Also examined were indentation load dependence of fracture toughness by the IF and the IS methods, and loading rate dependence of fracture toughness by the SEPB method.

Thirteen laboratories in Japan, Belgium, Canada, France, Germany, UK and CEC (JRC Petten) took part in the test. The results have been analysed by Japan Fine Ceramics Center (JFCC).

Round Robin Design

The ceramic materials used were gas-pressure sintered silicon nitride (GPSSN) and zirconia-alumina composite (ZAC).

These materials have a homogeneous structure. Twenty specimens for each material were sent to the participants. Dimensions of each specimen were 3 mm x 4 mm x 40 mm. One of the 4 mm width-sides was mirror-finished surface ground by a 2000 diamond wheel to improve the visibility of the Vickers impression and cracks on the IS and the IF specimens.

(1) IS method

The mirror-finished surface was indented by a Vickers hardness machine, using loads of 49 N and 294 N for GPSSN, and 98 N and 490 N for ZAC to estimate indentation load dependence. Then, three-point flexure strength of the indented specimen was measured over a 30 mm supporting span and with a 0.5 mm/min machine cross-head speed. Fracture toughness values were calculated by the formula of Chantikul et al¹.

(2) SEPB method

As a precrack starter, one 98 N indent was made for each GPSSN specimen and three in-line 196 N indents for each ZAC specimen. After making the precrack using a bridge-compression fixture, a dye penetrant mixed with acetone was used to improve the visibility of the precrack. A three-point flexure test with 16 mm supporting span was used to fracture the specimens. Two cross-head speeds (1 mm/min and 0.005 mm/min) were used to estimate loading rate dependence of fracture toughness.

(3) IF method

The indentation loads used were 98 N and 196 N (196 N and 294 N in Japan) for GPSSN specimens, and 294 N and 490 N for ZAC specimens.

Fracture toughness was calculated by two equations, Miyoshi et al², and Marshall and Evans³.

Results and Discussion

The full results will appear in VAMAS Technical Report No 9, which is due to be issued soon.

(1) IS method

Figure 1 shows indentation load dependence of fracture toughness measured by the IS method. These data show the quite small interlaboratory scatter in fracture toughness values in comparison with that observed by the other two techniques. Each participant's number is shown near the dot in the figure.

Results on annealed specimens after indentation measured by participant No 2 are also shown in the figure for reference. It means that the equation of Chantikul et al becomes invalid for the specimen after annealing treatment, because the analytical formula is based on the existence of the residual stress field induced by the indentation. It can also be considered that its applicability is doubtful at high temperature.

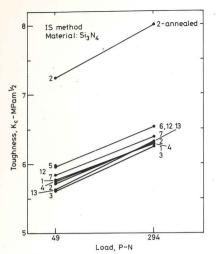


Figure 1. Indentation load dependence of fracture toughness determined by IS method

(2) SEPB method

There must have been some confusion in carrying out the SEPB test, because it was a new technique for the majority of the participants and it needed a special fixture. Nevertheless, many participants made the fixtures and evaluated the fracture toughness.

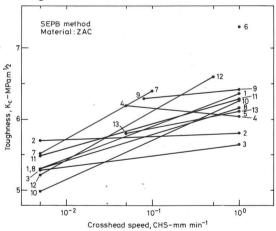


Figure 2. Loading rate dependence of the fracture toughness determined by SEPB method

Figure 2 shows the loading rate dependence of the fracture toughness for ZAC by the SEPB method. Relatively wide spread of results among laboratories suggests that the SEPB technique involved some difficulties, and that the workmanship of the fixture made by each participant considerably affects the results. The results obtained by the Japanese participants show small scatter because they are used to this method. Almost every datum in the figure increases with increasing cross-head speed. Subcritical crack growth by moisture is responsible for this behaviour, especially for ZAC. Therefore, the loading rate is considered to be one of the most important factors

in evaluating fracture toughness of ceramics containing some types of oxide, such as silica.

(3) IF method

The indentation load dependence of fracture toughness measured by the IF method is given in Figure 3 for ZAC. The fracture toughness was calculated by the formula of Miyoshi et al ². A slight decrease in fracture toughness with increasing load is seen. The extremely wide spread of the data might mean that it was difficult to make a mirror surface on the ZAC by diamond wheel grinding. As a result, it was difficult to detect the crack tips on the surface. It was found that some participants obtained the higher fracture toughness values with the higher standard deviations which might be caused by using a worn indenter. This means that the crack length created around the impression may depend on the condition of the diamond indenter used.

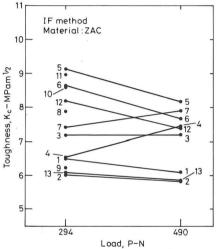


Figure 3. Loading rate dependence of the fracture toughness determined by SEPB method

Table 1 is the summary of the results. This table includes the fracture toughness values evaluated by the SEPB method with 1.0 mm/min CHS, the IF method with 196 N indentation for GPSSN and 294 N for ZAC, and the IS method with 294 N for GPSSN and 490 N for ZAC. The difference among the overall mean fracture toughness values obtained by these three techniques is rather small, compared with the scatter in results obtained by each laboratory.

Conclusions

The results of the VAMAS Fracture Toughness round robin test have been analysed to assess methods of measuring fracture toughness for advanced ceramics. It is concluded that:

- (1) Relatively wide scatter in the results obtained by the SEPB method is probably caused by difficulties in measurement technique.
- (2) The loading rate dependence of fracture toughness measured by the SEPB method is significant for ZAC.

- (3) The IF method gives the largest spread of results because of the difficulty in detecting the crack tips, especially in white (translucent) materials.
- (4) This IS method shows quite small interlaboratory scatter in comparison with the other two techniques. The analytical expression to calculate fracture toughness is only valid when the residual stress field is still present.
- (5) The fracture toughness measured by the IF and IS methods depends on the indentation load.

Acknowledgements

The authors would like to acknowledge the interest and effort all the participants provided for this project. We have received several useful comments from the participants which helped to make this report.

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- 2. MIYOSHI, T., SUGAWN, N. AND SUZZA, T. Proc JSMEEAS, 1985, 2489, 1-471.
- 3. MARSHALL, D. B. AND EVANS, A. G. J Am Ceram Soc, 1981, 64-12, 182.

Table 1 Summary of Results								
	Material GPSSN			Material ZAC				
Range of mean fracture	SEPB 1.0*	IF 196N**	IS 294N**	SEPB 1.0*	IF 294N**	IS 490N**		
toughness MPam ^{1/2}	5.16 - 6.42	5.14 - 6.42	6.26 - 6.54	5.65 - 6.56	6.02 - 9.15	6.69 - 7.96		
Range of std deviations	0.12 - 0.39	0.09 - 0.42	0.11 - 0.18	0.07 - 0.39	0.05 - 0.89	0.07 - 0.29		
Overall mean fracture toughness	5.66	5.75	6.37	6.14	7.36	7.41		
Std dev of mean	0.33	0.45	0.11	0.26	1.06	0.38		
Number of labs	10	12	8	10	13	8		

* Cross head speed,mm/min

** Indentation load, N

VAMAS Technical Reports

Several new VAMAS technical reports have been published and the following is a current up to date list of documents issued by VAMAS Technical Working Areas. Copies of the reports should be obtained from the appropriate TWA international chairman or from the VAMAS Secretariat.

- 1. "Results of the First Round Robin Comparison" TWA 1 - Wear Test Methods, 1987
- "Factual Materials Databanks: The Need for Standards" TWA 10 - Materials Databanks, 1987
- "The VAMAS Hardness Test Round Robin Ceramic Materials" TWA 3 - Ceramics, 1987
- "VAMAS Workshop Standards for Materials Databanks" TWA 10 - Materials Databanks, 1990
- "International Summary of the Classification of Advanced Ceramics Industrial Practices and Needs" TWA 14 - Classification of Advanced Ceramics, 1991
- "Significance of Data Evaluation Models in Material Databases" TWA 10 - Materials Databanks, 1990
- "Computational Models for Creep and Fatigue Data Analysis" TWA 10 - Materials Databanks, 1990
- "Guidelines for Conducting Hardness Tests on Advanced Ceramic Materials" TWA 3 - Ceramics, 1990
- 9. "Fracture Toughness Round Robin on Ceramics" TWA 3 - Ceramics, 1991
- "Interlaboratory Comparison Programme on Variable Weld Penetration -Synthesis Report" TWA 9 - Weld Characteristics, 1990

Technical Working Areas

Technical Working Area 1

WEAR TEST METHODS

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Objectives

- Develop internationally agreed wear test methodologies for advanced materials, initially ceramics and inorganic coatings
- Improve reproducibility and comparability of wear test methods
- Characterise wear behaviour of advanced materials.

The round robin interlaboratory comparisons, based on the first objective, resulted in several publications and standard test methods adopted by ASTM and DIN. A meeting is planned to discuss the future activities, based on the second objective. This meeting will be organised in conjunction with the International Wear of Materials Conference, April 7-11, 1991 in Orlando, Florida.

An important issue related to the characterisation of the wear behaviour and wear data is the minimum set of parameters that are needed to completely describe the materials and the tribological test conditions. A possible approach is to use a computerised database structure for assembling tribological data and evaluating the completeness of the data fields in a round robin exercise. This issue and other suggestions will be discussed and a future activity will be selected at the April meeting.

Technical Working Area 2 SURFACE CHEMICAL ANALYSIS

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Objectives

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

As must be clear from the increasing length of the six-monthly reports to the National Representatives of the TWA, the progress established in the latter half of the last decade continues to be enhanced. More projects are reaching completion and, through the modular structure of this programme we expect (i) the results to be fed back into new projects led from any Member State, and (ii) for new projects to arise in the original leading Member State. Projects that are now complete are the reference material work of project 1, the tests of method and materials in projects 16 and 17 and the standard format of project 10. The materials from projects 1 and 17 already appear in other projects and the standard format is being incorporated in both project 24 and the proposed project 29. This last example is a powerful illustration of the way the VAMAS umbrella can catalyse convergent activity in an effective and economic manner. As was clear in the initial proposal to set up this activity (M P Seah and C J Powell, NPL Report DMA(A)89 September 1984), the work as a whole is beyond the resources of any member state. Without the VAMAS umbrella each Member State must attempt to build their own "cathedral". With the VAMAS umbrella, parts may be made by many small groups which, with input from all of the Member States, are guaranteed to fit together to form a lasting structure.

Thus, with the standard data format (10) a major step forward was made. This provided a format which anyone writing data processing software could organise their programme to read. This then means that software could be exchanged and developed from laboratory to laboratory as each laboratory could produce their own data in the standard format. This represents a very efficient but flexible development . A major difficulty with the above scenario has been the rate at which the manufacturers have implemented the format for customers to output the data. Fortunately the initiative has been seized in the proposed project 29 to establish the File Format Translation System. Both this and the complementary project 24 to

establish conventions for spectral databases provide the essential basis in the short term for more intelligent spectral data analysis and, in the longer term, expert systems and the use of artificial intelligence structures in the study of processes at surfaces. The convergence of projects 10, 24 and 29, and the further project 30 to establish a Common Data Processing System provides an excellent foundation for our future developments in this area.

Projects 13, 14, 18 and 21 all involve the development of algorithms which need broad validation. All of these projects are progressing satisfactorily but all could benefit from being embedded in a general data processing package for more widespread validation as would be possible with project 30. Project 3, to determine composition-depth profiles from angularly resolved XPS measurements, also involves the development of algorithms but, at the present time is focused on establishing high quality experimental data with which to test models of varying complexity. This project was the topic of discussion at AVS in Toronto and, more generally, at QSA6 in London. Project 26, which extends the theory in this area using the Monte Carlo approach, is expected to provide further convergence between projects and to delineate the optimal experimental conditions for which the simpler quantification routines are valid.

It is pleasing to note that the projects involving reference materials and their data (projects 5, 6 and 8) are all proceeding strongly, since without these materials it is difficult to validate and calibrate many of the more difficult measurements. Reference materials are also involved in projects 20, 25 and 28 where a detailed analysis of binary metal systems across the phase diagram is possible. Two projects involving the difficult problems of insulators, projects 6 and 11, are progressing strongly with, on the one hand, polymeric reference materials being developed and, on the other, the understanding of charging and breakdown in defect-containing and defect-free ceramics is being addressed.

Some of our early projects involved instrumental calibration. In project 2, NPL has completed the studies for AES and XPS energy scales and these data are being homogenised with more recent data from NIST, both to be compatible with the result of the earlier round robin assessment. The intensity scales for AES are close to completion in project 9 and those for XPS currently being analysed in project 23. Project 22 shows the necessity for considering the calibration interval as well as the calibration itself.

The results of the above calibration work together with the procedures and infrastructural scientific work have, in the past, been formalised into standards by ASTM. ASTM now has a broad portfolio of standards from Committee E42 on surface analysis supported by a dozen or so active subcommittees. ASTM also has other committees such as E49 on Computerisation of Material Property Data which are also relevant to our work. Further standards are being developed by IUPAC. Following a Japanese initiative the TWA has considered the possible setting up of an ISO Technical Committee for SCA at its annual meeting of National Representatives. At the meeting there were presentations on how the ISO activity would operate from the Japanese representatives. It was concluded that the international aspects were very important but they may, at the present time, be most efficiently handled through ASTM if all National Representatives could be involved.

Details of projects will be found in the full reports distributed by National Representatives as follows:

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

VAMAS SCA PROJECT LIST (1 OCTOBER 1990) (* are for adoption)

Project No:

- 1. Development of thin oxide films as reference materials (M P Seah)
- 2. Development of calibration data for the energy scales of Auger-electron spectrometers (M P Seah, C J Powell)
- 3. Procedures for quantitative X-ray photoelectron spectroscopy (C J Powell, J E Fulghum for Phase 1)
- 4. Measurement of spatial resolution in AES
- 5. Development of reference materials prepared by ion implantation (W H Gries, D Gould)
- XPS intensity calibration and stabilisation with polymeric reference materials (C E Bryson)
- 7. Correction methods for backscattering in AES (J P Langeron)
- 8. Reference data for sputtering rates in oxides (H J Grabke)
- 9. Intercomparison of Auger-electron energy and intensity measurements (M P Seah)
- 10. Development of a standard data transfer format (W A Dench)
- 11. Multi-technique characterisation of vacancies in alumina (C Le Gressus)
- 12. Calibration of surface layers by nuclear reaction analysis (I V Mitchell)
- 13. Tests of algorithms for data processing in AES Factor analysis and intensity (P R Underhill)
- 14. (a) Tests of algorithms for background subtraction in XPS (S Tougaard)
 - (b) Tests of algorithms for background subtraction in AES (S Tougaard)

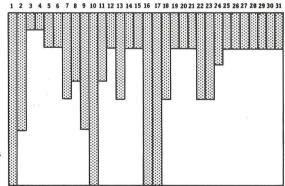
(c) Tests of algorithms for quantitative XPS by peak and peak background shape analysis (S Tougaard)

- 15. Evaluation of SNMS sensitivity factors (M Anderie)
- 16. Intercomparison of surface analysis of thin aluminium oxide films (P Marcus)
- 17. Quantitative AES of Au-Cu alloys (R Shimizu)
- 18. Evaluation of LOGIT, an algorithm for fitting-depth-profile data, for the measurement of interface widths of an NBS thin-film reference materials (J Fine)
- 19. Round robin SIMS study of impurities in GaAs crystals (S Kurosawa)

- 20. Round robin AES study of Co-Ni alloys (K Yoshihara)
- 21. Tests of algorithms for the analysis of multicomponent spectra in XPS (A F Carley)
- 22. Calibration of channel electron multiplier detection efficiency stabilities (M P Seah)
- 23. Absolute calibration of XPS instrument intensity scales (M P Seah)
- 24. Conventions for spectral data bases (R N Lee)
- 25. Quantitative XPS of Au-Cu alloys (K Yoshihara)
- 26. Theoretical assessment of escape depth (R Shimizu)
- 27. Multiline reference materials for differential AES intensity calibration (M P Seah)
- 28.* Quantitative XPS of Co-Ni alloys (K Yoshihara)
- 29.* Development of a File Format Translation System (K Yoshihara)
- 30.* Development of a Common Data Processing System for AES and XPS (K Yoshihara)
- 31.* Intercomparison of the Effects of AI on the Determination of Thin Oxide Film Thicknesses (P Marcus)

Stage of development of projects

- (i) Project announced
- (ii) National research
- (iii) International project plan
- (iv) International research
- (v) Data being analysed
- (vi) Report circulated
- (vii) Report analysed
- (viii) Work published
- (ix) RM/Data/Procedure in progress
- (x) RM etc. established



Technical Working Area 3 CERAMICS

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Objectives

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

Following the successful hardness testing round robin, a document has been prepared suggesting guidelines for the use of hardness measurements on ceramic materials. This is available as VAMAS Technical Report No. 8. The document has been prepared as a draft standard for immediate input to standards committees, and has already been circulated to CEN TC184, WG3 for discussion.

A final report on the Japan-organised fracture toughness test round robin, organised by Japan, has been prepared in draft form following receipt of 12 sets of results from participants. In summary, the round robin has compared three methods of testing advanced technical ceramics, namely the indentation strength method (IS), the single-edge pre-cracked beam method (SEPB), and the indentation fracture method (IF). By employing two ceramic materials with very homogeneous and reproducible microstructures, it has been shown possible to obtain very low standard deviations in the test results, and thus the differences in mean results between laboratories can be examined more meaningfully. While the majority of participants produced closely-grouped results, indicating that the procedures that had been defined were mostly adequate, some participants returned results significantly deviating from the mean. In the IF method, the difficulties of measuring crack lengths accurately by optical methods leads to the possibility of significant individual biases, and the results of this test had the greatest scatter between participants and the highest standard deviations for each participant. In the SEPB method, the design and operation of the "bridge" compression jig to propagate precracks from initial indentations was not simple and straightforward, and this, coupled with the difficulties of measuring crack length after the test led to some significant scatter in results. The simplest and most reproducible test was the IS method. The results also show that the equations used to calculate fracture toughness may not be completely adequate. In the IS method, toughness was unexpectedly found to be indentation load dependent. In the SEPB method, the average results were significantly lower than for the other techniques, possibly because the original indentation damage was not removed. Overall, the results give some clear indications as to the directions in which standardised test method development should proceed (See feature article in this Bulletin for more information).

The next phase of the TWA 3 programme will be a second fracture toughness round robin, this time examining a new V-notch beam technique, and comparing results obtained both at room temperature and high temperature with those using a chevron notch method.

Technical Working Area 4 POLYMER BLENDS

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Objectives

 To provide the technical basis for drafting standard test procedures for new, high performance polymer alloys and blends in 5 complementary technical areas:-Melt flows, Dynamic testing, Thermal properties, Morphology, Mechanical

1. Introduction

properties

The aim of the present Phase II of TWA 4 activities is to examine applicability of the test procedures identified in Phase I and to develop the technical basis for their adoption by ISO. To achieve these goals, the properties of several commercial polymer alloys need to be examined. The first of these is a compatibilised blend of polypropylene and poly-e-caprolactam, PP/PA-6, manufactured (and supplied free of charge) by Atochem under the name ORGALLOY R-6000 TM.

The 6th Annual Meeting of TWA 4 was held in Nice on 21-22.04.90. During the first day the test results on ORGALLOY R-6000 TM were presented by the technical area coordinators as well as by the representatives of "Groupement d'Intérêt Scientifique Alliages" (GIS-Alliages), a homologous organisation working in France toward similar goals and studying the same polymer alloy as TWA 4. The exchange of information was beneficial to both, allowing them to shorten the total evaluation process.

2. Technical Report on Characterisation of Orgalloy R-6000

2.1 Melt flow properties.

Dynamic as well as in the steady state shear fields at 230 $^{\circ}$ C and 250 $^{\circ}$ C were determined. The dynamic test results were highly repeatable with the standard error of measurements ±3%, similar to that calculated for single-phase melts. The inter-laboratory comparison showed good agreement within ±6%. The activation energy of flow at constant stress was determined as Ev = 60 kJ/mol. On the other hand, the capillary flow data were quite erratic with an error of ±10%. Furthermore, in capillary flow the shear stress at constant shear rate was found to be temperature independent. Similar discrepancy between the dynamic and steady state flow was observed for other immiscible polymer blends in this study, namely LLDPE/PC. The different responses originate in the flow modification of blend morphology. The

structure of the material under the dynamic small strains is different from that in the large strain capillary flow. In fact, the measurements are equivalent to testing two different specimens, eg polymer reinforced with spheres and that with fibres.

The elongational properties of molten ORGALLOY R-6000 were examined from both the entrance exit pressure drop in capillary flow and from direct tests in Rheometrics Extensional Rheometer. In both cases strain hardening was observed, yielding values more than one decade higher than those of the steady state elongational viscosity predicted by the Trouton rule: $h_E = 3h_0$. The source of the phenomenon is the PP drop deformability and strain induced crystallisation at the capillary entrance. Several papers on the melt flow of ORGALLOY R-6000 are being prepared for publication in scientific journals.

2.2 Viscoelastic properties.

Measurements were made in the solid state at T = 120 °C to + 160 °C and frequencies ϑ = 1 Hz to 90 Hz. The test specimens, having dimensions 25 mm x 12 mm x 2 mm, were cut out from either an extruded sheet or compression moulded plaques. Prior to testing, the materials were dried at 90 °C under dynamic vacuum. Systematic changes in the loss tensile modulus were observed for a drying period of seven days; three relaxation peaks observed at - 72 °C, 0 °C and 31 °C on drying shifted to - 40 °C, 0 °C and 64 °C. The kinetics of the first and the third peak displacement with drying time were found to be different, suggesting that the effect may be due not only to drying but also to more subtle changes in the crystalline morphology. The peaks were identified as respectively being due to internal friction of hydrated amide group, glass transition temperature (Tg) of PP and that of PA-6. The value of Tg agreed quite well with those reported in the literature. It should be noted that the slow drying process of the polymer blend originates in the basic structure of the material; drying of the homopolymers was found to be tenfold more rapid.

The 2 mm thick extruded sheets of ORGALLOY were also tested in the machine (MD) and transverse (TD) direction. Within the experimental error, the storage moduli of these specimens were the same. However, the loss moduli curves in MD (especially near Tg of PP) were higher and narrower than those in TD, indicating a sharper distribution of relaxation times in the principal orientation direction.

2.3 Impact properties.

Extruded, 2 mm thick, ORGALLOY R-6000 sheets were examined for impact performance at T = 25 °C and relative humidity RH = 50%. The hammer impact speed was 3 m/s. In all samples the fracture was brittle. The fracture energy calculated by the J G Williams method was Gc = 5.9 kJ/m² and 10 kJ/m², for MD and TD specimens, respectively. Since Tg of PA-6 matrix is above the test temperature the brittleness was expected. The literature value of Gc for PA-6, Gc = 8.2 kJ/m², is comparable to those found for the blend.

In another series of tests, using injection moulded plaques, the specimen bars 10 mm x 10 mm x 100 mm were machined out and tested in a Charpy configuration. In good agreement with results of the preceding tests the critical fracture energy parameters were calculated as $Gc = 6.7 \text{ J/m}^2$ and $Kc = 3.67 \text{ MN/m}^{1.5}$.

2.4 Tensile tests.

Tests were conducted on specimens cut in MD and TD directions from extruded, 2 mm thick ORGALLOY sheet. The tensile and yield strength decreased with temperature, while the ultimate elongation increased (see Table 1). For TD specimens, the latter parameter was significantly lower than that for the MD ones. The difference was particularly large at low temperatures (70 vs. 355% at RT). The TD results were quite erratic.

	Ambient	60 °C	100 °C
1. Machine Direction			
Tensile Strength (MPa)	39.4	41.8	32.4
Ultimate Elongation (%)	355	388	401
Yield Strength (MPa)	34.1	25.0	15.4
2. Transverse Direction			
Tensile Strength (MPa)	36.4	32	24.4
Ultimate Elongation (%)	70	367	391
Yield Strength (MPa)	34.6	24.0	14.3

TABLE 1. Tensile Test Data for ORGALLOY 2 mm thick extruded sheet

2.5 Thermal properties.

The three polymers were analysed in a differential scanning calorimeter. There was good agreement between data from Germany and Japan in spite of the difficulties in determining Tg of PA-6. Two peaks in ORGALLOY R-6000 were found to correspond to the melting point of PP ($T_m = 165.6$ °C) and PA ($T_m = 223.2$ °C). By comparing the heat of fusion of the three polymers it was discovered that in ORGALLOY there is a significant amount of excess materials which does not participate in the crystallisation process. Furthermore, on scanning it increased from 13% to 21% from the first and the third scan, respectively. The samples were extracted with solvents leaving the non-crystallisable material behind. The infrared spectroscopy identified it as an amorphous mixture of PA-6 with PP acting as compatibiliser.

2.6 Morphology.

Little work has been done in this area. The ORGALLOY R-6000 is made of PA-6 matrix and dispersed PP. In the "as received" state, PP is dispersed in a form of small spherical particles (number average diameter $d_n = 1.11 \pm 0.63 \mu m$) in the PA-6 matrix. The morphology changes during processing. At high output rate extrusion the drops coalesce and elongate to form fibres. Owing to the shear fractionation, the outer skin of extrudate is nearly pure PP.

ORGALLOY R-6000 testing will continue in all VAMAS countries for another year.

3. Recent Events

3.1 New blends.

During the 6th Annual Meeting in Nice a mixture of polyphenylene ether with polyamide was selected as the second commercial blend to be examined by TWA 4. Supply of one ton of this material was offered by BASF in Germany and Allied Signal in the USA. The companies also offered help in specimen preparation. In view of the significant cost involved these generous offers provide a welcome sign of recognition for the role TWA 4 plays in development of sound test procedures for polymer blends.

3.2 Future of TWA 4.

In response to an enquiry from the VAMAS Steering Committee the following questions were circulated for discussion within the participating national VAMAS organisations:

- 1. What are the goals of the TWA 4?
- 2. Are specific tests for polymer blends really needed?
- 3. Why should the TWA 4 work be carried out in five different technical areas?
- 4. Is there any sense in testing several blends instead of one?
- 5. Why are funds needed to accomplish the VAMAS goals?
- 6. What is being planned for the future?

The replies were surprisingly alike:

- 1. The aim of TWA 4 is to provide the technical basis for drafting standard test procedures for new, high performance polymer alloys and blends.
- 2. In general, the blend morphology changes with processing procedure. For this reason most of the tests developed for single phase polymeric materials cannot be applied. Those which can must be identified and the type of blends to which they can be applied must be defined.
- 3. Industry requires test procedures not in one, but in several technical areas. Furthermore, the pre-standardisation work on polymer blends involves research, ie, there is a need for data from different areas of expertise to understand material behaviour in order to determine validity of the proposed test methods.
- 4. Owing to the variety of polymer blends, applicability of methods developed on a single blend may not be valid for others. If the standard test procedure is to have general use its applicability must be verified on several types of these materials.
- 5. There was a consensus that (i) the funds must originate not from a centralised VAMAS organisation but rather from within each member country, and (ii) the support should be provided for long term (weeks, months or years) work on VAMAS project, material supplies, shipping costs and travel expenses.
- 6. During the coming year an effort will be made to finish work on ORGALLOY R-6000 and then examine two other commercial blends. Starting in 1992, an emphasis will be placed on morphology and mechanical properties.

Thus, there is a clear consensus where the TWA on polymer blends should be going. Within the next two years we should wind down activities on thermal properties, melt rheology and dynamic testing of solids. The results obtained within these three technical areas of activity on two types of blends will be put to ISO as "New Work Items". Two years from now, the work within TWA 4 should concentrate on interrelations between morphology and mechanical properties.

3.3 Organisational changes.

Drs Shaul Aharoni and L Choplin have resigned from their respective positions. They are replaced, respectively, by Dr Murali K Akkapeddi from Allied-Signal as the USA representative and by Dr Morris G Rogers as coordinator of the technical area on melt rheology.

3.4 TWA 4 meetings.

National meetings were held in France on 5.07.90 and in the UK on 16.11.90.

Technical Working Area 5 POLYMER COMPOSITES

Prof C Bathias, Conservatoire Nationale des Arts et Métiers Dept of Materials Engineering, 292 rue Saint Martin, 75141, Paris, France Tel: +33 1 40272322 Fax: +33 1 42719329

Objectives

- To assess and refine fracture toughness measurements for delamination crack growth
- To develop test procedures, data presentation and failure criteria for fatigue of continuous fibre composites using flexural and tensile test conditions
- To develop creep test procedures for continuous multidirectional composites

Fatigue Round Robin

Testing by participants continues for this programme. The results for the materials distributed first should be available shortly for inclusion in an interim report. Delays in the supply of some laminates for specimen preparation and a modification to the recommended test procedures will delay the final report by a few months. The test procedure has been modified to use prescribed stress levels based on the ultimate strength and fatigue strength values measured by the coordinator at NPL. Difficulties are being experienced by some laboratories who have recorded values for the ultimate tensile strength which are below the highest recommended fatigue stress levels. Additional tensile specimens prepared by the programme coordinator are being dispatched in order to study this problem further.

Delamination Test Methods

The second phase of this programme concerns delamination crack growth under fatigue loading conditions. Tests are being performed under both Mode I and Mode II conditions. Materials used in phase 1 that are being re-tested under these fatigue test conditions include unidirectional glass fibre/epoxy, woven glass fibre/epoxy (both thin and thick specimen geometries) and two grades of carbon fibre/epoxy.

The effect on the crack growth rate (da/dN - where a = crack length (mm), N = cycle) of the test frequency and the R ratio (minimum/maximum) are being studied. A report has been published covering the results from one French laboratory; other laboratories are still conducting their tests. The French results suggest that a frequency change from 5 Hz to 10 Hz had no effect but that for a constant maximum energy release rate the growth rate increases with increase in R ratio.

Technical Working Area 6

SUPERCONDUCTING AND CRYOGENIC STRUCTURAL MATERIALS

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Objectives

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

The standardisation of measurement methods in both superconducting and cryogenic structural materials is a key for the progress of superconducting technology which plays an important role for energy saving and development of new technologies.

The first round robin test on critical current, Ic, measurement in three different Nb_3Sn wires with 24 participant laboratories from nine countries was completed in 1988. The strain caused by the difference in thermal expansion coefficient between Nb_3Sn and the measurement mandrel was found to be the main origin of scatter in Ic. The second round robin test using the same measurement mandrel and under more specified conditions is now in progress.

The first round robin test on AC loss measurement in filamentary Nb-Ti wires was completed in 1989. The results have been analysed, and the second round robin test using seven Nb-Ti wires with a systematic difference in filament diameters will be started in the near future. The measurement will be focused on the AC susceptibility method and the vibrating sample magnetometer method.

In the area of cryogenic structural materials, the round robin test on tensile measurement at 4.2 K on 316 LN and YUS 170 steels was completed in 1988. The data were reasonably consistent. Most of the scatter in data was associated with the method of strain measurement. Thus, a round robin test on the use of strain gauges at cryogenic temperatures was initiated in 1989. Meanwhile, the round robin test on fracture toughness measurement at 4.2 K using the same steels was completed in 1989. The factors affecting the scatter in data were deduced. Recently, the second round robin tests on tensile and fracture toughness measurements at 4.2 K using Ti alloy (Ti-5AI-2.5 Sn) under more refined conditions have been started.

The 7th TWA meeting will be held in June 1991 at the time of the International Cryogenic Materials Conference, where the intermediate results of the second round

robin tests on both superconducting and cryogenic structural materials will be discussed. If the VAMAS cooperation of this TWA is renewed in 1992, it will be a good time to start the standardisation of measurement techniques for high-Tc oxide superconductors as well as composite cryogenic structural materials, since by that time sufficient homogeneous samples for round robin tests will become available in these new materials.

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Technical Working Area 7 BIOENGINEERING MATERIALS

Dr T Tateishi, Mechanical Engineering Laboratory 1-2 Namiki, Tsukuba-si, Ibaraki 305, Japan Tel: +81 298 54 2509 Fax: +81 298 54 2549

Objectives

 To establish internationally agreed procedures for biocompatibility testing of bioengineering materials using cell culture methods

1. Background

Significance of in vitro biocompatibility evaluation

Many kinds of materials have been implanted into the human body, replacing joints, bones, teeth, vessels, etc. It is natural to test strictly 'biocompatibility' of these materials, because they must be safe for human use.

Test materials are generally implanted into animals for the biocompatibility test. However, this kind of test (*in vivo* test) is expensive, time-consuming and not quantitative. Moreover, the *in vivo* test is becoming increasingly undesirable and socially unacceptable.

On the contrary, *in vitro* tests such as tests by a cell culture method give quantitative evaluation with a short period of experiments, although it could not perfectly reflect *in vivo* conditions. Therefore, the *in vitro* test is expected to be promoted for biocompatibility evaluation and to be ameliorated so that it could minimise the use of *in vivo* tests.

Difference between procedures in VAMAS/TWA 7 and in ISO/TC194

There are two concepts concerning biocompatibility in the cell culture method, namely cytotoxicity and cytocompatibility. Cytotoxicity and cytocompatibility must be classified into two separate categories. Cytotoxicity is concerned mainly with assessing harmful effects of toxic chemicals released from materials while cytocompatibility is concerned with assessing compatibility associated with electrochemical, micromorphological and physical characteristics of material surfaces.

The procedure in the ISO/TC194 is focused on the cytotoxicity, evaluating toxicity of leaching chemicals from test materials. The procedure in the VAMAS/TWA 7 is focused on the cytocompatibility, wholly evaluating compatibility (including toxicity) of test materials by making cultured cells directly contacting test materials.

The TWA 7 continues contact with Dr Nakamura, the Japanese representative in ISO/TC194 so that the procedures will not overlap each other.

2. International Collaboration

At this moment, the following researchers are ready to participate in the international round robin tests: Professor Buddy Clark (USA) Dr George Stookey (co-participant) (USA) Professor D F Williams (UK) Professor Pascal Christel (France) Professor Arturo Pizzoferranto (Italy) Dr Tetsuya Tateishi (Japan)

3. Test Plans

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3.1 Cell growth and cell adhesion tests

The TWA 7 has prepared tentative procedures of cell growth and cell adhesion tests by a cell culture method. According to these procedures, preliminary round robin tests in Japan were already completed in 1989 by a sub-group* of the TWA 7. The round robin tests resulted in proving that the procedures give quantitative and reproducible results.

The TWA 7 is to promote international round robin tests, first of all, according to these procedures.

It is inevitable, for what we called biomaterials, to contact with living cells *in vivo*. However, according to the position where the biomaterials are used, they are required to have either of two different characteristics, one of which is not to make cells adhere to the surfaces such as a frictional surface of an artificial hip joint, and the other of which is to make cells actively interact with the surfaces such as a surface of artificial hip joint's stem.

Molecular level researches have elucidated that living cells adhere to materials, with an interaction between fibronectins absorbed to material's surfaces and firbronectin receptors on the cell membrane, and that the interaction influences the cytoskelton by transmembrane control.

In order to measure adhesion strength, a viscometric method has been developed by which shear stress is loaded on cells. On the other hand, vertical stress to the material's surfaces tries to be directly loaded on cells in this procedure. The feature of this procedure is to be able to quantify the adhesion strength itself, because vertical stress is directly loaded to the mass of cells adherent to material's surfaces.

(*)

Dr Tetsuya Tateishi Agency of Industrial Science and Technology Professor Hideki Aoki Tokyo Medical and Dental University Professor Yosito Ikada Kyoto University Professor Masaaki Nakamura

Osaka Dental University

Specimens

Biomaterials such as hydroxyapatite, alumina, zirconia are sintered into plates (diameter: 30 mm, thickness: 1 mm) so as to be put into cell culture dishes (2500, Corning, USA). The surfaces of the specimens were polished so that the surface roughness (R_a) was to be 0.1.

Apparatus for loading vertical stress

By attaching an apparatus made of aluminium to a centrifuge, vertical stress to the specimens' surfaces is directly loaded on the cultured cells adherent to the surfaces.

Cell culture

L-929 derived from mouse connective tissue and MG-63 derived from human osteosarcoma are cultured under the condition of 37 °C, 5% CO₂ concentration in air. The culture medium is prepared with supplementing Eagle's MEM with 10% foetal bovine serum, 2 mmol L-glutamine and 18 mM NaHCO₃. The cells are subcultured once a week and seeded on the specimens after four days from the subculture.

3.2 Cell morphology test

The cell culture method is a popular one for biocompatibility test of implant materials, and it has several merits in comparison with *in vivo* implant method, such as short test span, good reproducibility and economical running cost. In that method there are, however, many parameters to evaluate the biocompatibility, and it is still disputable which is relatively suitable for the evaluation of the biocompatibility. In these parameters, cell morphology could be considered to represent the interaction between material surface and cell membrane, and to represent the cell's viability. The cell morphology had been difficult to be treated quantitatively, though other parameters are easily done. In this procedure, cell morphology is tried to be treated quantitatively in using so-called shape factors by means of the image analysing method.

Specimens

Biomaterials such as hydroxyapatite, alumina, zirconia are sintered into plates (diameter: 30 mm, thickness: 1 mm) so as to be put into cell culture dishes (2500, Corning, USA). The surfaces of the specimens were polished so that the surface roughness (R_a) was to be 0.1.

Cell culture

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L929 cells are seeded into dishes so that the cell density would be 0.2×10^4 cells/cm². After 3 days' incubation, the cells are immobilised overnight at 4 °C with 2.5% glutaraldehyde solution. The immobilised cells are stained with 5% Giemza solution for cell morphology tests.

Image analysis

The images of cells through a polarising microscope are input with a video camera for low illumination intensity (min 0.3 lux). The input images are A/D-converted into 512 x 512 pixels at 256 grey levels by an image processor. The A/D-converted images are

processed into binary images by selecting an appropriate threshold value so that black areas represent cells and white areas represent material surfaces. Then the values such as area, perimeter and maximum diameter of a cell are calculated from the binary images.

4. Material Supply

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All the test materials used in the above-mentioned tests are to be supplied by Japanese industries.

Technical Working Area 8 HOT SALT CORROSION RESISTANCE

Dr T B Gibbons, Division of Materials Metrology NPL, Teddington, Middlesex, UK, TW11 0LW Tel: +44 81 943 6026 Fax: +44 81 943 2989

Objectives

 To develop unified procedures for assessing the hot salt corrosion resistance of superalloys in burner-rig tests

The testpieces for the international collaboration, which is being carried out to probe the validity of the Guideline document, have been distributed to all participants. The materials involved are:

- IN738
- IN738 coated with RT22, platinum alumina
- Rene 80
- Rene 80 coated with ATD2, CoCrAIY.

The intercomparison involves 500 h exposures in defined conditions at each of two temperatures namely 700 °C and 900 °C.

The participants include the leading manufacturers and users of superalloys for gas turbine application in the USA, Europe and Japan.

Technical Working Area 10 MATERIALS DATABANKS

Dr J Westbrook, SCI-Tech Knowledge Systems,133 Saratoga Road NY 12302, USA H Kröckel, CEC JRC, Petten, ZG-1755, The Netherlands Tel: +31 2246 5208 Fax: +31 2246 1002

Objectives

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

Chairmanship: Dr Rumble, NIST, has resigned as co-chairman of TWA 10; his task will be taken over by Dr Westbrook, SCI-Tech Knowledge Systems. Dr Rumble has co-chaired TWA 10 since its establishment in 1985 and given strong leadership to its activities which have generated momentum towards standardisation due to his verve and persistence. TWA 10 owes him much of its success.

Status Summary: Activities are in a transition from Phase 2 to Phase 3 where two main subjects will be addressed:

(1) Materials Data Interchange

In Phase 2 an International Workshop on this subject was held at Rolls Royce plc (September 1989); in Phase 3 it is planned to compare Materials Data Interchange Methods in a number of inter-institutional data exchange tests. Progress so far is slower than anticipated. Some fundamental problems have arisen in the work of ASTM Committee E-49 which should produce a first round of data exchange tests preceding the VAMAS activity and using, among others, formats agreed by the Rolls Royce Workshop. These tests have not yet been possible, and there may be a need for some redefinition of the ASTM and VAMAS activities.

(2) Materials for Data Evaluation, Analysis and Models

In Phase 2 an Interlaboratory Comparison of Data Evaluation Methods used in materials databanks was organised by NRIM; in Phase 3 the assembly of an Inventory of Methods/Models for Materials Data Analysis and Evaluation is planned. The New Materials Centre (NMC) of Japan has produced a first plan for this activity in co-operation with NRIM.

(3) Inventory of Materials Designation Systems

This work in Phase 2, undertaken by K W Reynard, UK, has shown much progress; a first draft of the main part is under review in TWA 10. Distribution of and follow-up to this extensive compilation need definition in the near future.

Technical Working Area 11 CREEP CRACK GROWTH

Dr T B Gibbons, Division of Materials Metrology NPL, Teddington, Middlesex, UK, TW11 0LW Tel: +44 81 943 6026 Fax: +44 81 943 2989

Objectives

 To develop a unified approach to the measurement and interpretation of creep crack growth data

The procedure agreed at the end of the Phase I of this project on measurement and interpretation of creep crack growth in the steady-state have been formalised in a draft standard which is currently being reviewed. A second version of the draft is being considered by the members of the VAMAS group and has been forwarded to the terminology committee of ASTM. A revised third draft will then be produced and will be submitted to the ASTM voting procedure. A full standard should emerge in early 1992 provided the current rate of progress can be maintained.

In the second phase of the project the initiation and early stages of crack growth are being considered. A model which enables these initial stages of crack growth to be predicted is being evaluated by members of the group. Efforts are being made to obtain additional creep-growth data, particularly for low-ductility material, to enable bounds of applicability for the correlating parameter C* to be defined. Results from Japanese participants on large section specimens will provide a basis discriminating between plane stress and plane strain deformation conditions.

The next coordination meeting of the group is scheduled for 27 and 28 July 1991 in Tokyo, immediately preceding ICM6.

Technical Working Area 12

EFFICIENT TEST PROCEDURES FOR POLYMER PROPERTIES

R P Brown, Rapra Technology Ltd., Shawbury, SY4 4NR, UK, Tel: +44 0939 250383 Fax: +44 0939 251118

Objectives

- To provide support for standardisation activities in the characterisation of long term properties of viscoelastic polymer materials
- To produce a guide for accelerated durability tests
- To consider the role of standard reference materials in accelerated durability testing

The broad objectives of the TWA are to provide support to standardisation activities in the characterisation of the long term properties of viscoelastic polymer materials. Relevant properties include creep, stress relaxation, fatigue and durability in aggressive environments. In order to avoid prohibitive amounts of testing over long time scales it is common practice to make use of extrapolation, interpolation, interpolation, interconversion of property data or accelerated tests. Many of the procedures are at best approximate and they often lack standardisation.

A specific area currently being addressed by TWA 12 is that of the durability of polymers in aggressive environments such as heat, light, and liquid chemicals with particular reference to accelerated tests.

Current work is addressing two topics:-

- Provision of a general guide to the use of accelerated durability tests (ADT) and is being coordinated by R P Brown, Rapra Technology Ltd., Shawbury, SY4 4NR, UK, and could, in due course, provide the basis for a standard.
- Consideration of the role of standard reference materials (SRM) in ADT led by G Zerlaut, DSET Laboratories, Box 1850, Black Canyon State, Phoenix, Arizona, USA. It will provide one or more guides to the use of SRM in ADT.

A broad survey amongst VAMAS participants evaluated their priorities with respect to the different aspects of the general TWA context (i.e. creep, stress relation, durability, etc.) and was responsible for the choice of durability as the current specific area.

A detailed survey examined the availability, status and use in VAMAS countries of standard tests for ADT and this led to the selection of the two current work topics identified above. A paper giving broadly the results of the survey has been accepted for publication in "Polymer Testing".

A study of the use of SRMs in ADT has been produced by G Zerlaut and considerable progress has been made in the production of a comprehensive guide in the use of ADT.

Future plans for TWA 12 suggest that attention should remain in the area of ADT as surveys indicated that other aspects, such as creep and stress-relaxation, are inappropriate for VAMAS action at the present time.

Technical Working Area 13 LOW CYCLE FATIGUE

Dr D Gould, CEC, rue de la Loi 200, B-1049, Bruxelles, Belgium Tel: +32 2 235 9313 Fax: +32 2 235 8046

Objectives

 To identify those aspects of testing procedure that significantly affect the reproducibility of the results of low cycle fatigue tests at high temperatures

Evaluation of Low Cycle Fatigue Test Data in the BCR/VAMAS Intercomparison Programme

The objective of the LCF programme was to establish a set of high temperature data representative of accepted testing practice and, subsequently, to identify the significant aspects of testing procedure affecting the repeatability and reproducibility as a basis for defining a standard test procedure. The test conditions, including strain ranges, strain rate, waveshape, temperature and limits to testpiece surface finish, were specified in guidelines circulated to the participants. However, each laboratory used its normal test methods, ie testpiece form, extensometry, testpiece manufacture, test machine and recording equipment.

In view of the potential influence of material characteristics on LCF life three classes of alloy were selected for testing according to their cyclic strain behaviour. These were defined as strain-hardening (AISI 316L steel at 550 °C), strain-softening (either 9CrMo steel or IN718 alloy at 550 °C) or stable (Nimonic 101 at 850 °C). Twenty-one data sets were supplied for AISI 316L steel, 16 for 9CrMo steel, 12 for IN718 and 12 for Nimonic 101.

The results indicate that interlaboratory differences in life were particularly significant in the case of high strength superalloys tested at low strain ranges. The type of testpiece and extensometry were significant factors in some cases. In particular, methods involving testpieces with transition radii within the measured length or those employing diametral extensometry gave results that tended to lie at the extremes of the scatterband more frequently than those obtained using smooth parallel testpieces with side-contact axial extensometry.

Repeatability and reproducibility also appeared to be influenced by materials characteristics, in particular the strength of the alloy and the proportion of plastic strain exhibited at the test conditions. The general level of spread in the results could not be attributed to any single major testing parameter, nor was there any evidence of material variability. The differences in life attributable to different definitions of failure were minor compared with the general interlaboratory variation.

Currently further attempts are being made to re-analyse the data either in its entirety or sub-sets to try to understand the causes of the scatter.

Technical Working Area 14

THE TECHNICAL BASIS FOR A UNIFIED CLASSIFICATION SYSTEM FOR ADVANCED CERAMICS

S J Schneider, NIST, Gaithersburg, Md 20899, USA Tel: +1 301 975 5657 Fax: +1 301 926 8349

Objectives

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

An explicit objective of VAMAS is the transfer of its pre-standards research to standards organisations and the community at large, in a way that facilitates and assures the translation of the precursor activities through to the adaptation and final standardisation by standards bodies. Early-on coupling of pre-standards work with actual standards development has long been identified as the preferred and most efficient route to an approved and recognised standard. The process is much like, and parallels the coupling required in the innovation process of technology (ie the intimate linkage of basic, applied and development research leading to a commercial product).

Each of the VAMAS projects handles the coupling task in different ways. For VAMAS TWA 14 (Classification of Advanced Ceramics) the methodology chosen follows three pathways, each of which is direct and overt. First, the makeup of the working membership was orchestrated to have representation by members of major standards bodies (eg ASTM, DIN, BSI, CEN, JSI etc) and by industrial spokes-persons (eg USACA, Verband der Kerasmischen Industrie, Groupisol, private companies). To this were added government and university representatives with the end result being an international working group that touches and interfaces with all the interested organisations needed in the standardisation process, including ISO. In effect all the institutional links were built in from the start.

Second, community awareness is a mandated objective of TWA 14. Many key tasks undertaken are carried out through a public forum process, as for instance the conduction of an international survey and the organisation of an open workshop. All primary outputs have or will be publicly presented at professional meetings and published in the open literature, with special distribution to organisations and people who needed to know and who will be the most likely to utilise the results. For this a 1200 person international list of who's who in ceramics and standards has been compiled. The third and perhaps most crucial step has been to establish mechanisms and lay the groundwork for direct standards action by organisations like the CEN secretariats and the ASTM Committees, C28 on Advanced Ceramics and E49 on Computerisation of Materials Property Data. The basic matrix-type classification scheme currently under development once finalised, will be introduced, explained and promoted for adaptation by appropriate national standards interest and action groups (ie for the US, USACA and ASTM C28/E49). Action and directions taken by these groups is of their own choosing, but nonetheless the relationships have been established with guidance provided by TWA 14.

The transfer process has started. Already Committee C28 and the EC secretariat concerned with classification have formally endorsed the VAMAS effort and await the results, at which time appropriate action will be taken and the consensus process started towards a classification standard for advanced ceramics.

VAMAS Calendar					
7th Annual TWA 4 (Polymer Blends) Meeting Hamilton, Ontario	25 April 1991				
VAMAS Steering Committee Meeting Montreal, Canada	22-24 May 1991				
7th Annual TWA 6 (Superconductivity & Cryogenic Structural Materials), International Cryogenic Materials Conference University of Alabama, Huntsville, Alabama, USA	e, June 1991				
TWA 11 Meeting preceding ICM6 Tokyo 4 th Topical Conference on Quantitative Surface Analysis prior to 38th National Symposium of AVS at Seattle,	27-28 July 1991				
Washington, USA	8-10 November 1991				

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