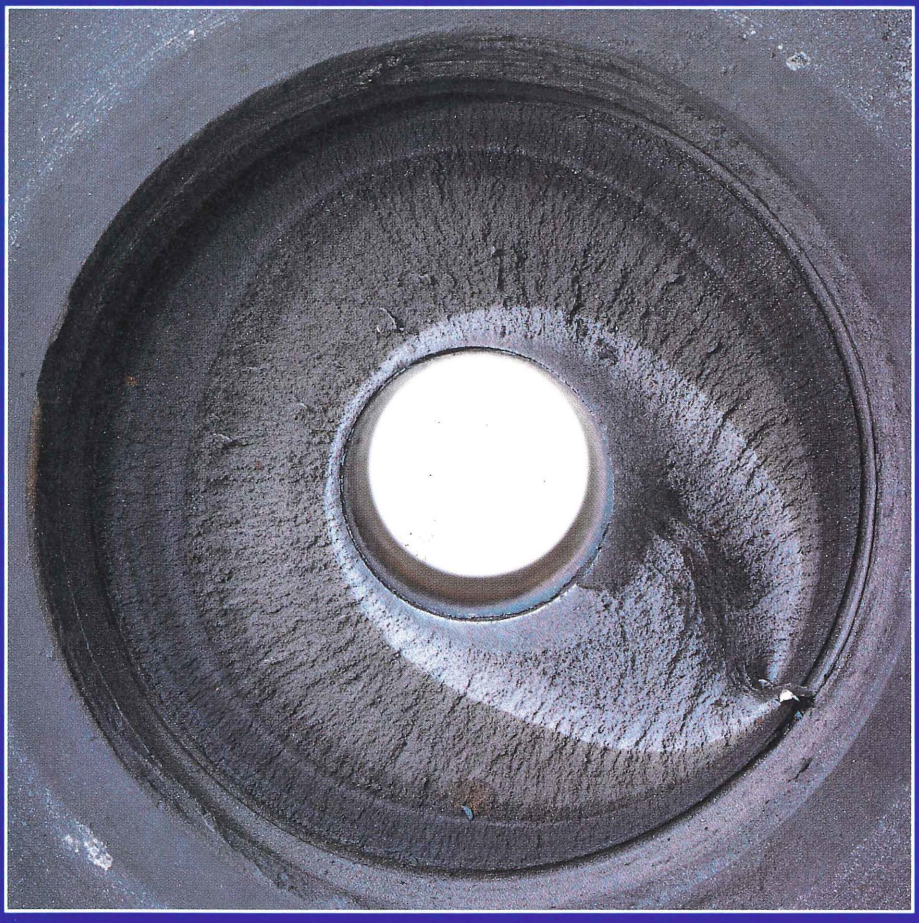
VAMAS



BULLETIN NO. 11
January 1990

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

VAMAS

The Versailles Project on Advanced Materials and Standards (VAMAS) supports trade in high technology products through international collaborative projects aimed at providing the technical basis for drafting codes of practice and specifications for advanced materials. The scope of the collaboration embraces all agreed aspects of enabling science and technology - data bases, 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.

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 and F R Germany, Italy, Japan, the UK, the USA, and the Commission of European Communities are each represented on its Steering Committee.

Cover: Fracture surface of bolt after full scale laboratory test.
See feature article by T B Gibbons.

Photograph by courtesy of Dr S Holdsworth, GEC Alstham Turbine Generators Ltd, Rugby, UK

VAMAS

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SECRETARIAT TRANSFER

VAMAS is jointly led by the UK and the USA, each providing the Secretariat alternately for three year periods. Dr Lyle H Schwartz (Chairman) and Dr Bruce Steiner (Secretary) from NIST (formerly NBS) have represented the USA in guiding VAMAS through the years 1986-1989. At the tenth Steering Committee meeting at NPL in June 1989, the reins were transferred to the UK for the years leading to 1992. This period is going to be an especially important one for VAMAS because 1992 marks a review point for the Memorandum of Understanding under which VAMAS operates. The new Chairman is Dr Kamal Hossain who heads Materials Research at the National Physical Laboratory and the Secretary is Dr Bryan Roebuck also from NPL.

This is a good opportunity to put on record the debt owed by the VAMAS organisation to Drs Schwartz and Steiner of NIST for providing a strong momentum that has stimulated development and steady growth of our activities in the last three years. Both have shown a commitment and enthusiasm to VAMAS which should provide a bedrock for the future. They have put in a tremendous amount of personal effort and dedication to promote the VAMAS concept and a glance at the VAMAS publication list shows how their labours have borne fruit. Increasingly strong ties have been developed with national and international standards bodies such as ISO, ASTM, DIN and BSI. Lyle H Schwartz and Bruce Steiner saw this as an important item on the agenda and through proper encouragement and their persuasive skills VAMAS has made substantial progress on this front. Finally it is clear even at this stage that VAMAS will continue to benefit from the application of the knowledge and experience that they have acquired in providing the Secretariat for 1986 - 1989.

Publication Guidelines

As the Technical Working Areas mature in their various activities the need for their output to be effectively disseminated in reports, papers and notes is growing. The VAMAS Aims and Organisation document lists guidelines for dealing with publications which individual Technical Working Area Chairmen have responsibility for putting into practice. Clearly they are only guidelines and Chairmen have some flexibility in their operation. To reinforce the process the following paragraphs from the Aims and Organisation document are repeated below:

"6. The output of each contributor in a Working Area will be freely communicated to all other contributors in the area and there will be free exchange of information at all meetings of Technical Working Parties. The information and reports resulting from VAMAS cooperative activities shall however remain confidential to the contributors in a Working Area until such time as the contributors and the Technical Working Party Chairman agree to wider dissemination.

7. It is the responsibility of all participating organisations to ensure that they will be able to conform to the principles of free exchange or, when this is not entirely possible due, for example, to commercial confidentiality or licensing arrangements, to inform other actual or potential contributors from the outset of any restrictions. In the event of restrictions being unacceptable, the Chairman has the right to refuse participation.

8. Contributors will be free to publish results of their own work provided that results of other VAMAS contributors and details of the VAMAS programme are not included. Articles based on collaborative work may be published in the normal manner, subject to approval by the Working Party Chairman to ensure that the authorship is appropriate and that results of cooperative effort are not prematurely disclosed. Standards documents will be developed and disseminated through existing national and international standards bodies. All published reports of VAMAS sponsored activities should include an acknowledgement indicating the role of VAMAS and, unless otherwise requested, should reference the origin of the information and samples. Copies of all such reports should be forwarded to the Working Party Chairman for transmittal to the Secretary of the Steering Committee."

The guidelines do not include comment on timetables but it is worth noting that the Surface Chemical Analysis Working Area has adopted the following phrase to avoid potential embarrassment:

"All manuscripts describing VAMAS-SCA projects or results should be submitted to the SCA Chairman who will distribute copies to all national representatives with a covering letter asking that any comments be supplied within six weeks. In the absence of compelling argument against publication, publication will be approved by the SCA chairman. Approval for publication may, however, be conditional on specified revisions being made to the manuscript."

In addition to technical papers organised by individuals and Technical Working Partners, use is also made of full VAMAS reports. The VAMAS Bulletin can also provide a medium for communication of project precis through technical notes and feature articles. Three full reports have now been issued, and a further one is near to print, with the following titles:

VAMAS Report 1 "Results of the first Round Robin Comparison"
TWA1 - Wear Test Methods, 1987.

VAMAS Report 2 "Factual Materials Databanks: The Need for Standards"
TWA10 - Materials Databanks, 1987.

VAMAS Report 3 "The VAMAS Hardness Test Round Robin on Ceramics Materials"
TWA3 - Ceramics, 1988.

VAMAS Report 4 "VAMAS Workshop - Standards for Materials Databanks"
TWA10 - Materials Databanks, to be published 1990.

• Feature Article •

MEASUREMENT AND INTERPRETATION OF CREEP CRACK GROWTH IN A CrMoV STEEL

A VAMAS COOPERATIVE PROJECT
by T B GIBBONS
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Introduction

In the design and operation of high temperature plant it is important to characterise the defect sensitivity of the materials used in critical load-bearing applications. Design stresses must take account of the maximum defect size that can be tolerated and, should a crack develop during service, estimates of growth rate will influence inspection intervals and remanent life prediction. In order to make these estimates, measurements of crack growth are made on laboratory testpieces and the data obtained are interpreted in the context of service conditions. Various techniques have been developed for measurement and interpretation of crack growth data and the need for a more harmonised approach has been widely recognised. Thus in the mid 1980's various groups around the world initiated intercomparison programmes with the overall aim in each case of establishing a common approach which would lead to a standardised procedure. The VAMAS initiative provided an internationally recognised framework within which these independent and parallel activities could be unified.

Participation and Organisation

The purpose of the VAMAS activity was to facilitate collaboration between the various international groups involved in research on creep crack growth measurements. The Participating Groups are given in Table 1 along with the names of the National Coordinators in each case. The overall objective was to develop a compatible body of technical information which would serve as a basis for national or international standards and Codes of Practice. While several different materials have been investigated by various groups, the first phase of the VAMAS activity which is now nearing completion was concerned with the behaviour of a CrMoV steel which was common to all participants. Guidelines issued for the ASTM programme were also used by the EGF participants and this provided a core activity with a common experimental approach.

Experimental Procedures

For the most part, compact tension (CT) specimens, some with side grooves, were used although some participants examined double-edge notched (DEN) or centre notched (CEN) specimens. Test temperatures were 550 °C for the European participants and 538 °C and 594 °C for the Japanese and ASTM Groups. Crack growth was measured by potential-drop (either AC or DC) methods and precautions were taken to monitor potential changes due to modification of the microstructure during isothermal ageing.

Parameters to Characterise Crack Tip Behaviour

The stress and strain distributions at the tip of a growing creep crack can be complex and depend on the rate of stress redistribution in relation to the rate of crack growth and the creep conditions ahead of the crack. In some situations where complete stress redistribution has occurred at the crack tip and the region ahead of the crack is in steady state creep, the conditions at the crack tip can best be represented by the parameter C*. There are several computational methods which can be used to derive C* and in Figure 1, the data obtained by the EGF Group for CrMoV steel at 550 ^{o}C are displayed. There is clearly a large amount of variability in the data and this is partly due to the variation in C* as a result of the different methods of calculation used by the various participants.

As a consequence of extensive discussion within the VAMAS Project, a common procedure for calculating C* for conditions of complete stress redistribution and steady state creep ahead of the crack has been agreed. The relationship is given by

$$C^* = \frac{P\dot{\Delta}}{B_n b} \cdot \frac{n}{n+1} \cdot \eta$$

where P is the applied load

$\dot{\Delta}$ is the creep displacement rate at the crack tip

B_n is the net specimen width

b is the length of the uncracked ligament

n is the stress component of the steady state creep rate

η is a geometric factor which for CT-type specimens is approximately 2.

An important point is that the equation agreed for C* is consistent with that given in ASTM E813 for J viz:

$$J = \frac{P\Delta}{B_n b} \cdot \frac{n}{n+1} \cdot \eta$$

In this case n is the strain hardening coefficient, and Δ is plastic displacement.

Results of Crack Growth Rate Measurements on a CrMoV steel

When the data obtained by the EGF Group were reassessed using the unified method for calculation of C* the variability in the results was very much reduced as indicated in Figure 2. This was particularly true for the linear part of the plot of C* as a function of crack growth rate but, in the early stages of crack growth, the so-called "tails", substantial scatter remained. However elimination of results which did not meet a validity criterion viz:

creep displacement rate ($\dot{\Delta}_c$) >elastic displacement rate ($\dot{\Delta}_{el}$)

reduced the variability but did not remove the "tails" entirely (Figure 3).

The results shown in Figure 2 include data from various sizes and types of specimen and it is clear that specimen dimensions have little effect on crack growth behaviour. A similar pattern of results was obtained from the intercomparisons carried out in USA and Japan and all three sets of data are shown plotted on a similar basis in Figure 4. The level of consistency between the various sets of data, bearing in mind that test conditions were not identical in every case, is encouraging.

Overall the results confirm that a good measure of agreement can be obtained provided a unified procedure is used for calculating the correlating parameter C*.

The experience gained from this international intercomparison has been used to develop a draft Standard Test Procedure for creep crack growth measurement in conditions where a steady-state creep situation exists at the crack tip. When an agreed draft has been finalised within the VAMAS Group the document will be available to national and international standards bodies for adoption as a standard for creep crack growth measurement.

Outstanding Issues

While substantial progress has been made in unifying the procedures for obtaining and interpreting crack growth data, several major factors remain incompletely understood.

Currently there is no satisfactory technique for analysing the early stages of crack growth, ie the so-called "tails" on the plots of growth rate as a function of C*, which may represent a substantial portion of the life of the testpiece. Also, there are no well-defined limits to the applicability of the C* parameter; for example, in brittle materials where crack growth will proceed more rapidly than stress redistribution can occur, C* is unlikely to be the appropriate correlating parameter. Thus there is a need to examine the limits of applicability of C* and of the other parameters which are used to describe stress and strain conditions at the crack tip.

The ultimate objective is to relate laboratory data to the behaviour of components and this remains a major outstanding area of investigation. In particular it is necessary to define procedures for determining crack tip parameters in complex components.

In order to make progress in these remaining areas a second phase of the VAMAS programme has been planned.

Table 1 Participating Groups and Co-ordinators

(i)	ASTM Committee E24-04; Dr A Saxena, Georgia Institute of Technology.
(ii)	European Group on Fracture (EGF); Task Group 1: Dr T Hollstein, Fraunhofer Institute, Freiburg, Professor G A Webster, Imperial College.
(iii)	Japanese Society for Promotion of Science (JSPS); Committee 129: Professor T Yokobori, Sendai.
(iv)	National Research Institute (Japan); Dr C Tanaka.
(v)	French National Programme; Dr P Balladon, UNIREC.
(vi)	UK Atomic Energy Authority (Interatom); Dr J Curbishley.

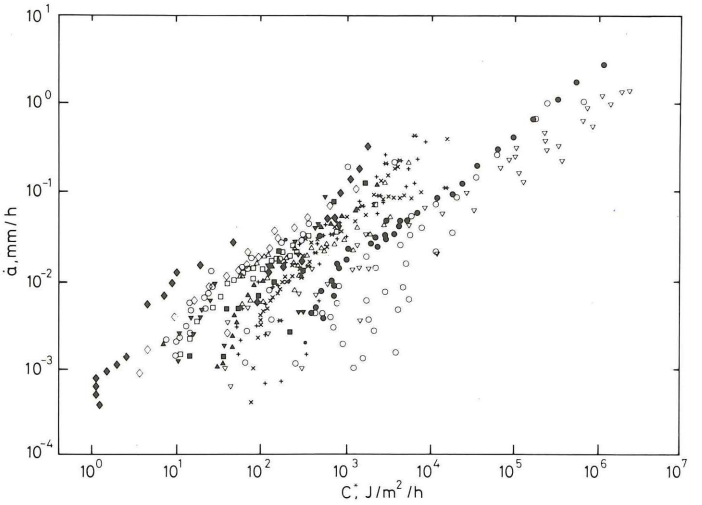

Fig 1. Crack growth rate as a function of C* for Cr Mo V steel at 550°C. EGF data showing variability arising from different computational methods for C*

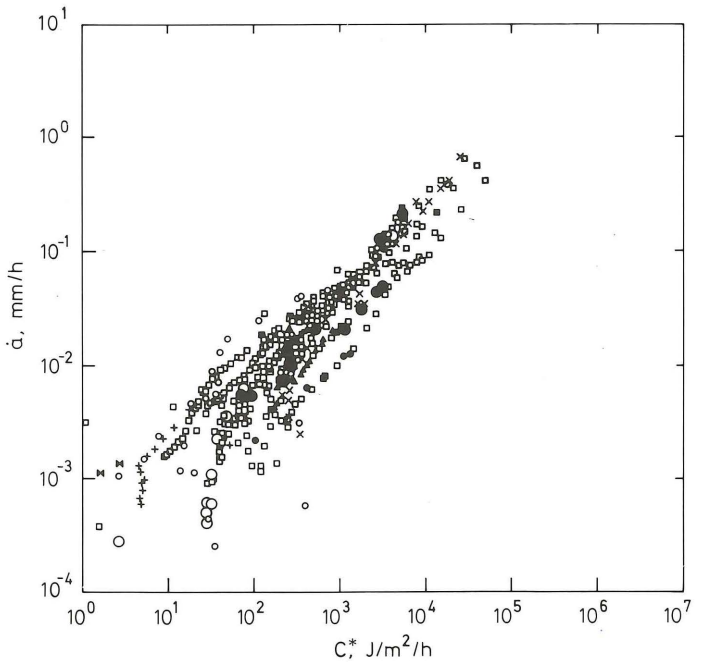

Fig 2. Data from figure 1 recalculated using harmonised computational method for C* as given by equation 1.

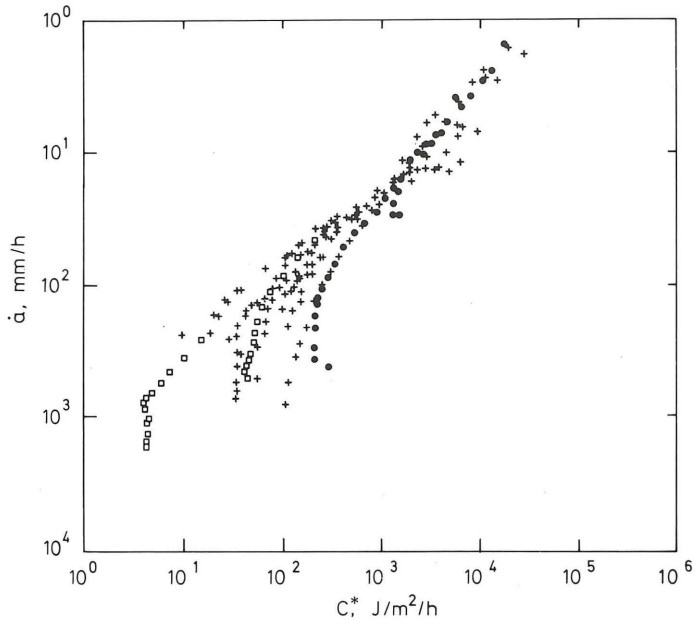

Fig.3. Results (for different specimen geometries) of application of validity criterion to early stages of crack growth: "tails" remain. CrMoV steel at 550°C

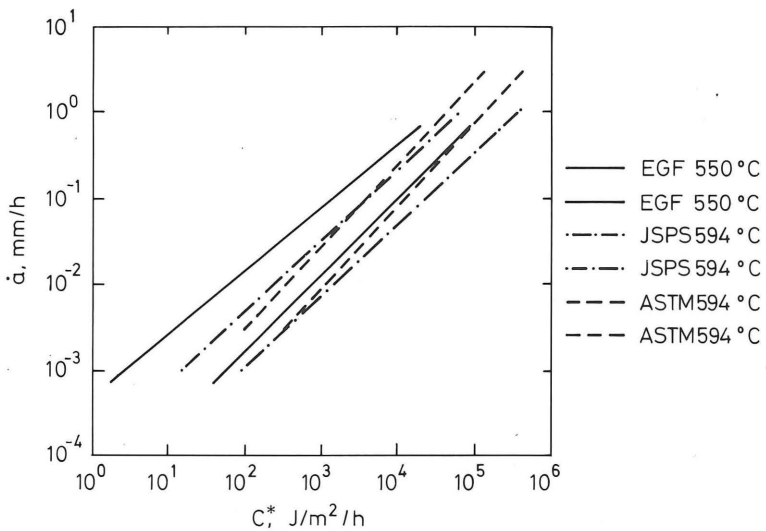

Fig 4. Comparison of data obtained by EGF at 550°C with results from ASTM and JSPS at 594°C.

• Technical Working Areas •

Technical Working Area 1

WEAR TEST METHODS

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As reported in Bulletin 10 a second round robin on the dry sliding wear of combinations of α-Al_2O_3 ceramic, Si_3N_4 ceramic and AISI 52100 steel has been performed, evaluated and the results compiled to a paper. After review by members of the TWA the paper has been accepted by the international journal WEAR. In supplementary technical comments, from the experience of TWA groups in the USA, it was noted that in lubricated, low load tests the reproducibility of the results was increased and the magnitude of wear decreased significantly.

A questionnaire has been sent out to members of the TWA to elicit views on continuation projects. Professor Czichos presented a preliminary analysis of the replies at the recent VAMAS Steering Committee Meeting at NPL in June 1989. Most interest from the first replies to the questionnaires were in the following areas:

(a) effect of hard coatings

(b) effect of specimen geometries

(c) more details on the effect of speed and load.

Technical Working Area 2

SURFACE CHEMICAL ANALYSIS

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During this period we have seen a number of notable developments and welcome a number of new projects as shown in the project list of Table 1. Project 6 is re-launched with a significant momentum by Dr Bryson to consider calibration and stabilisation with polymeric reference materials in XPS. Project 22, to consider channel electron multiplier stabilities, has evolved as a direct result of the responses to the round robin in project 9. Project 23 has also evolved from the same source as a result of the development of an absolutely calibrated electron spectrometer to set up that round robin. Both projects will involve activities in all Member States except that project 22 will be for those already responding to project 9.

Project 24, to set up the conventions for spectral data bases, is extremely important and, if done correctly, will have a major impact on the analytical community in the 1990s. This is the next logical step between project 10 and the ultimate goal of the unattended instrument managed by an expert system. Project 25 is a natural extension of project 17 and illustrates a particularly important point discussed below.

In the development of reference materials considerable work is invested. If we can use the same material for more than one project the advantages are, (i) the material is already partly characterised and so the new project proceeds more rapidly, and (ii) in the new work the consistency of interpretation can clarify or add to the characterisation of the first project (or it may even show up faults in the first project which would not have come to light otherwise). A multiplicity of projects bearing on one material has, therefore, genuine advantages. This now means that the creator of the first project must ensure a large supply of homogeneous material, or there must be a simple, reliable way of creating the material, or there must be a simple method of very precisely relating any new material to the old stock. We should try, as far as possible, to ensure that material supply does not limit our activity since many of the projects depend on materials. It is particularly pleasing, in this respect, to see a commercial supplier of materials involved in our programme in project 6 since this is likely to guarantee a supply of materials whilst they are of use.

This brings me to a second point I wish to emphasise here and this concerns traceability. If we wish to improve accuracy we must be aware of traceability. The interaction diagram, Fig 1, shows how projects may interact together and derive their traceability. Several of the projects have very careful traceability, with defined errors, to an accurate measure of, for instance, voltage (project 2) or quantity (project 1) or integrated charge (project 5). Wherever possible the accuracy of traceable data should be confirmed by more than one laboratory and this will be done in project 2. The reason for this is that it is genuinely difficult to define errors accurately. Again,algorithms should be related to first principles

continued on page 13

INTERACTION DIAGRAM FOR VAMAS SCA TWA PROJECTS

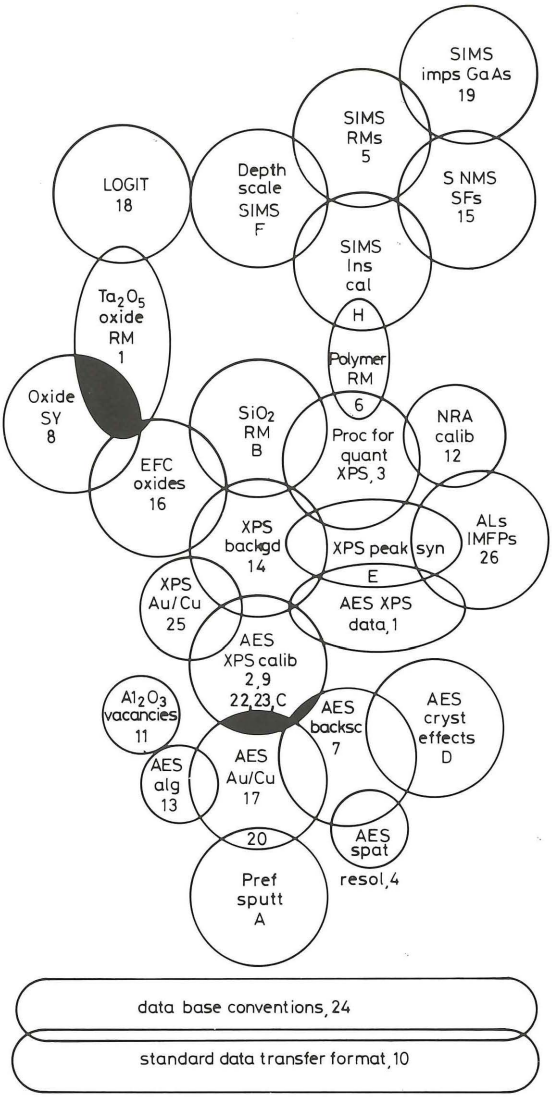

Fig 1. The interaction diagram for SCA projects. The overlaps show the interactions to be established before projects are complete. For simplicity every interaction is not shown. The shaded interactions show some of those where co-ordinating research is in progress

TABLE 1

VAMAS SCA PROJECT LIST (1 SEPTEMBER 1989)

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)
4. Measurement of spatial resolution in AES
5. Development of reference materials prepared by ion implantation (W H Gries, D Gould)
6. 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. Multitechnique characterisation of vacancies in alumina (C Le Gressus)
12. Calibration of surface layers by nuclear reaction analysis (J A Davies)
13. Tests of algorithms for data processing in AES - Factor analysis and intensity (P R Underhill)
14. Tests of algorithms for background subtraction in XPS (S Tougaard)
15. Evaluation of SIMS sensitivity factors (M Anderle)
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 sputter-depth-profile data, for the measurement of interface widths of an NBS thin-film reference material (J Fine)
19. Round Robin SNMS 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 (G C Smith)
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)

rather than parametric fits and this is illustrated very well in project 14. Algorithms should be validated as far as possible, as illustrated by projects 13 and 21. It may not always be possible to meet all these ideals and then we must resort to, for instance, homogeneous batches of material where the traceability can be established *a posteriori* as the science and measurement bases develop.

The last of the new projects, project 26, concerns the very important problem of escape depths. Here a new theoretical treatment will be developed. A major problem here has always been the experimental data to test the theories and since the start of the TWA a reliable experimental method has been sought to provide data, without success.

Turning now to the earlier projects not already mentioned, new material has been generated in project 1 and is being accurately related to the older material. The overall quality appears to be somewhat better. In project 2 detailed reports on the calibration and the results of the (40 laboratory) round robin have been circulated to all respondees and National Representatives for comment. During the next period NPL and NIST will prepare a joint recommendation for the calibration of energy scales. Project 3, which is crucial to much XPS interpretation, is now begining to move forward with material characterised for other work. NPL will begin to consider accuracy here. Project 4 unfortunately still remains unstarted as the original project leader could not afford the time. Project 5 is now progressing on several fronts but we would expect progress to be slow. This project has a greater metrology base than many others and clear attempts are being made to define accuracies properly. This is very difficult work but, if accurate traceability can be established, a small core set of materials will allow a considerable improvement in the accuracy of many other extant materials.

Project 7 is moving ahead well with some commonality to the reference procedures in project 9. A round robin is currently in progress. In project 8 reference materials are presently being established and it may be that these could also be useful to projects 3, 12 and 14, which in turn would help to further characterise the materials. The round robin for project 9 was conducted simultaneously with that for project 2. The results show that an excellent calibration may be achieved which, for some instruments, may be at a level better than 1% for intensities in the direct mode. Reports have been written and will be mailed in the near future. The Standard Data Transfer Format in project 10 is being implemented by manufacturers and is incorporated in Projects 20 and 24. Project 11 appears to be closing through lack of financial support, there being few people wanting to work on insulators. This is very sad since most practical analysts are forced to work on insulators whether they like it or not and they do need help. I would like National Representatives to consider if they can set up any projects to solve some of the practical problems here. In project 12 we welcome the involvement of Dr Mitchell since nuclear beam techniques offer one of the few traceable routes to absolute measurement.

Several projects involve validation and testing of software. Ross Underhill clearly had problems in mailing discs to participants in project 13 but we are pleased to note that the LOGIT mailing in project 18 has proceeded satisfactorily (to NPL at least) without the discs being impounded, scrambled or bent. Projects 14 and 21 are still proceeding at the research stage.

In project 15 those involved will all meet together at SIMS VII. It may be possible to bring projects 5 and 19 together here to their mutual benefit. The final report for project 16 has been circulated to National Representatives and the authors are to be congratulated on a particularly sound piece of work.

As mentioned earlier in project 18 the reference material and software have been mailed to participants and data will be collated in the new year.

Projects 19 and 20 invite new participants and it would be useful if the leaders in projects allied to these could be involved to ensure the cross fertilisation between projects.

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

Canada = 12, 13

France = 7, 11, 16

FRG = 5, 8

Italy = 15

Japan = 17, 19, 20, 25, 26

UK = 1, 2, 9, 10, 22, 23

USA = 2, 3, 6, 18, 24

CEC = 14

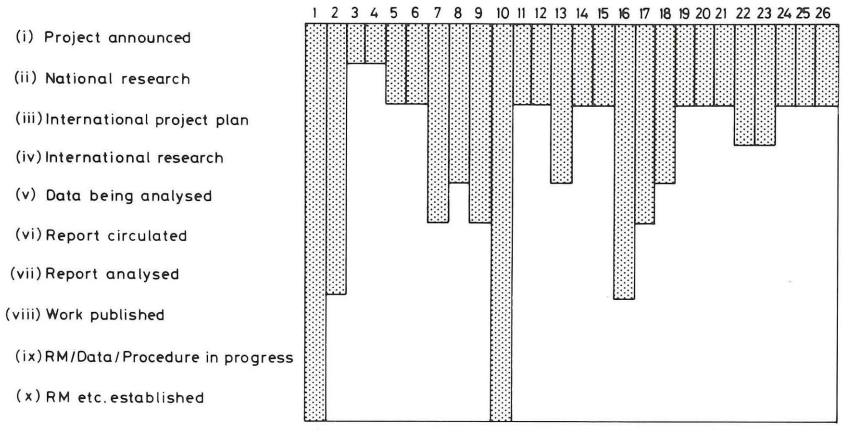

Fig 2. The stage of progress of the individual SCA projects. The length of the bar shows the stage reached

Possible or Proposed Multilateral Activities

Proposed SCA involvement in current CEC projects (Gould)

(A) Reproducibility of ion beam sputtering (Seah)
(B) Granular and planar SiO_2 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, Viefhaus)

Projects Under Development

(F) Development and test of a procedure to establish a 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)

Future International Meetings involving the VAMAS SCA TWA Work

Quantitative Surface Analysis, QSA 6, Royal National Hotel, London
UK. 12-16 November 1990

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The round robin on toughness measurements by the "single edge precracked beam" method is nearly completed. Dr Awaji (Japan Fine Ceramics Centre in Nagoya, Japan) is now collecting the data produced by the round robin and will soon be in a position to draw conclusions from this exercise. The round robin is examining three methods for reproducibility among laboratories. The three techniques are:

(i) The indentation strength method (fracturing testpieces containing defined indentation cracks)

(ii) The single-edge pre-cracked beam method (fracturing beams with straight-through pre-cracks produced by the "bridge" method from indentation flaws)

(iii) The indentation fracture method (measuring crack lengths produced directly by indentation)

The materials being tested are a sintered silicon nitride and a zirconia toughened alumina, both supplied by NTK Ceramics Ltd of Nagoya, Japan.

There is also to be a session on standardisation issues at the 7th CIMTEC (European Ceramic Conference) at Montecatini Terme, Italy, June 1990 which will include papers on VAMAS topics.

POLYMER BLENDS

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The goal of the Technical Working Party on Polymer Blends (TWP-PB) is to provide a technical basis for drafting standard test procedures for polymer alloys and blends. Round robin tests are being carried out in five technical areas: (1) melt flow, (2) dynamic testing of solids, (3) thermal analysis, (4) morphology, and (5) mechanical properties.

In Phase I of the program the viability of several test methods was examined. The report on thermal analysis, published in Polymer Engineering and Science 28, 1077 (1988), provided a basis for submitting to ISO TC-61 (Plastics) a new work item "Thermal Analysis of Immiscible Polymer Alloys and Blends (T550)", approved last June by majority vote of the ISO member organisations. At present a report on the dynamic testing of solids is being published while other reports (on melt rheology, morphology and mechanical properties) are in various stages of preparation. It is expected that other "new work items" will originate from these reports.

The purpose of Phase II is to examine the applicability of the test procedures identified during Phase I, using commercial blends containing combinations of three types of polymers; rubbery, glassy and crystalline. The first blend (donated to the program by Atochem) is Orgalloy$^{(R)}$ R6000, a crystalline/crystalline compatibilised blend of polypropylene (PP) dispersed in poly-e-caprolactam (PA). The homopolymers (PP from Japan Petrochemical and PA from Allied-Signal Inc.) are also available as reference materials to obtain additional reference information.

PRELIMINARY RESULTS OF ORGALLOY$^{(R)}$ TESTING

1. Thermal analysis and related characterisation methods

TABLE 1

No.	*Parameter*	*PA*	*PP*	*Blend*
1	Glass transition temp.,T_g (^oC)	42.1	3.0	
2	Onset melting point, (^oC)	214.4	155.2	
3	Peak melting point, T_m(^oC)	223.2$^{(a)}$	165.6$^{(b)}$	
4	Heat of melting, (J/g)	-46.1	-32.7	
5	Decomposition temp.,T_d (^oC)	450.0	449.5	456.5(456.0(b))
6	Decomposition temp. in air, (^oC)	280		
7	Density, (kg/m^3)	1208	937	1030(1029(c)) (1097(d))

Notes:

(a) Tm of neat PA-6 and i-PP is 223 ^oC and 165 ^oC respectively;

(b) PA/PP mixtures;

(c) calculated from volume additivity for PA:PP = 40:60;

(d) calculated from density additivity for PA:PP = 60:40.

There were no unexpected complications in measuring the thermal properties of Orgalloy$^{(R)}$. The DSC measurements were performed according to ISO standard 3146. The procedure was found to be satisfactory for the neat polymers as well as for the LLDPE/PC (Linear low density polyethylene/polycarbonate) and PA/PP blends.

2. Morphology

Microscopy of the residue after a 5-minute treatment in dilute formic acid revealed that the blend has PA as a continuous phase in which PP is dispersed as spherical inclusions with an average diameter d = 1.11 ± 0.63 μm. Analysis by FTIR confirmed the 40:60 composition. After subtraction of the neat polymer spectra from that of the blend there were no distinct peaks to identify the compatibiliser. One possible explanation can be that poly(propylene-g-amine) is playing that role.

3. Melt Flow

The specimens cut from 2mm thick extruded sheets (supplied by Atochem) were predried at 100 ^oC for 24 hours under dynamic vacuum. The test was conducted at 225 ^oC and 250 ^oC in parallel plate geometry. The samples were loaded at 250 ^oC then, when necessary, the temperature was lowered to 225 ^oC. The strain sweep at frequency, ω = 10 rad/s and temperature, T = 225 ^oC indicated linear viscoelastic behaviour within the range of strain $\delta \leq 25\%$. Consequently, the tests were conducted at $\delta \leq 15\%$. The time sweep showed that even at the lower temperature of 225 ^oC, the system was not stable. The variation of the rheological function suggested an increase of PA molecular weight.

The frequency sweep indicated normal viscoelastic behaviour expected for a thermoplastic, except for the presence of an apparent yield stress (at low frequency), originating from the multiphase nature of the blend. The agreement of interlaboratory test

data was within 10% (see Figs. 1-3). It was found that because of the two-phase nature of blends the preparation of specimens and test procedure must be more rigorous than that for single phase polymers. Similarly as for LLDPE/PC the Orgalloy(R) showed different behaviour under large strain capillary flow from that under the small strain in dynamic tests. The large strains induced changes in the morphology reflected in a difference in the flow performance as illustrated in Figure 4.

4. Dynamic Behaviour in Solid State

The dynamic spectra of Orgalloy(R) were found to contain only the characteristic elements of PA and PP, without any evidence for a compatibiliser. The time-temperature superposition principle was found to be valid. The horizontal shift factor a_T followed Arrhenius' equation with $\Delta H = 0.36$, 0.50 and 1.2 MJ/mol in three ranges of temperature.

5. Mechanical Behaviour

Only stress-strain data at room temperature, on the 2mm thick extruded specimens, have been obtained so far. The ASTM procedure was followed without apparent complications. The material was found to be sensitive to orientation and to the strain rate (especially as far as the yield stress was concerned). There are continuous difficulties with moulding an adequate supply of thicker specimens ($t \leq 6$ mm) for the fracture tests. At the present moment 5 kg of plaques for preliminary tests have been moulded for the program by Atochem. Without good specimens the work is limited to the use of diverse "in house" methods of drop impact tests with results dramatically illustrating a need for a well founded international procedure in this field.

Two other commercial polymer blends; high impact polystyrene and polybutyleneterephthalate/polycarbonate soon will be available for testing.

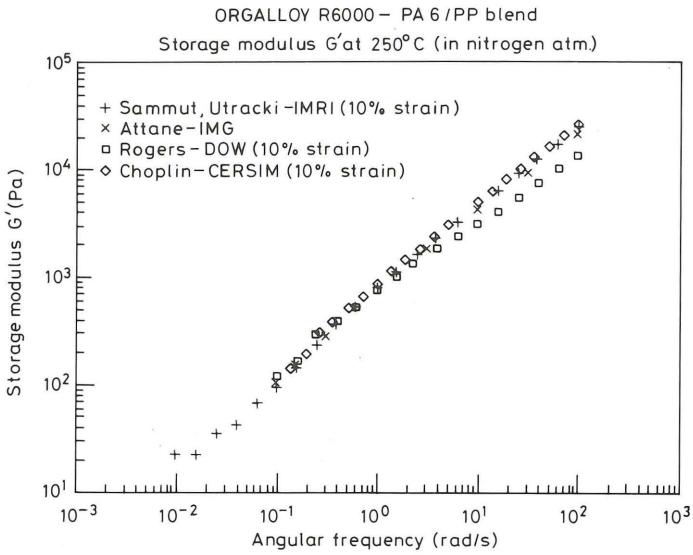

Fig1. Storage modulus G′ at 250 °C in nitrogen for ORGALLOY R6000

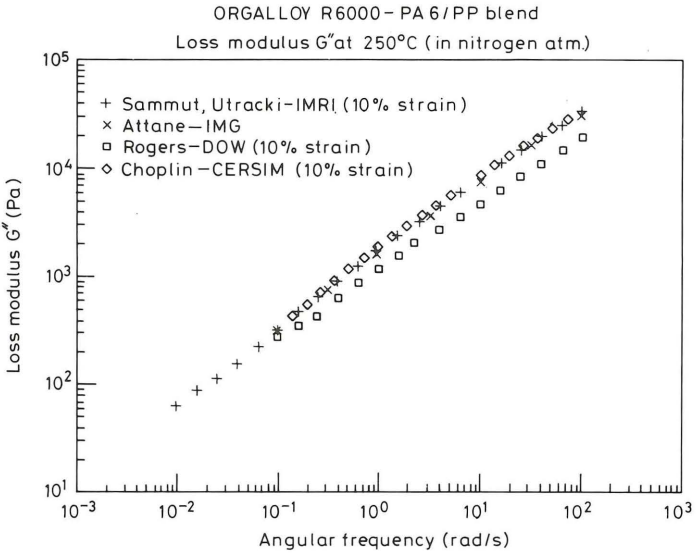

Fig 2. Loss modulus G″ at 250 °C in nitrogen for ORGALLOY R6000

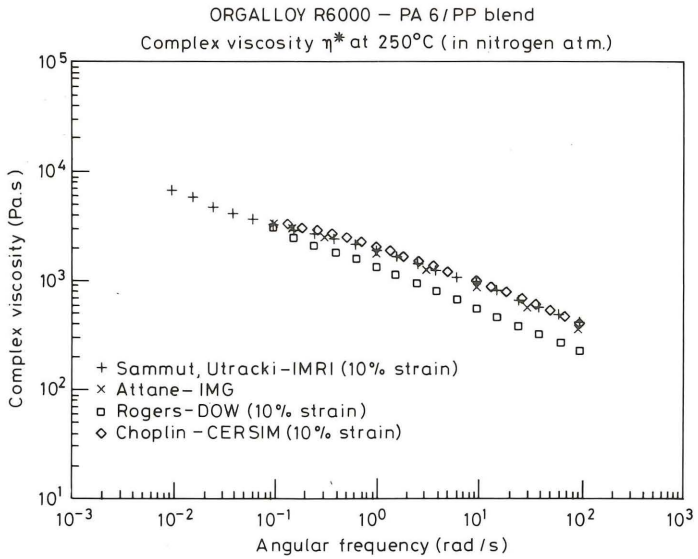

Fig 3. Complex viscosity of ORGALLOY R6000 at 250 °C in nitrogen

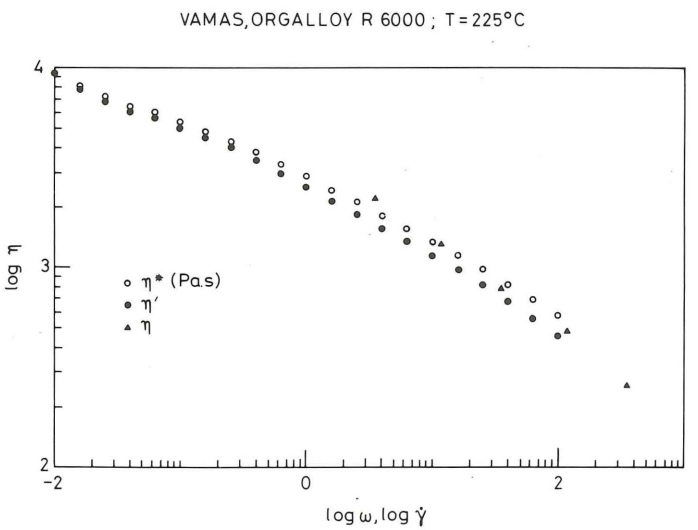

Fig 4. A comparison of viscosity measurements on ORGALLOY R6000 plotted against strain rate and frequency

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Of the three work areas, delamination, fatigue and creep, it is the fatigue round robin that has made most progress during the last period.

Fatigue Round-Robin

The first phase of this round-robin concentrates on a comparison of displacement controlled, flexural tests and load controlled, tension tests, with load controlled, flexure tests supporting the comparison. The planned test programme took into account the inability of most experimentalists to undertake both test modes, as axial tests are normally conducted on high capacity servo-hydraulic test machines whereas displacement flexure tests are conducted on low capacity machines often driven by cams or levers using small capacity electric motors. In addition there are no currently agreed international standards for the equivalent static specimens (ie ultimate failure tests at a slow rate).

The planned programme contains for both loading modes a core test programme and specification; and an optional development test programme for those able to study further the detailed aspects of the test method. For the tensile test programme these development tests could include effect of specimen width, end-tab design and tab adhesive. Similarly for flexure development tests could include, 3 v 4 point loading, span-to-depth ratio, support and loading roller diameter. The core test specimen and loading specification are based on the most advanced National or European standards.

The programme therefore contains the two individual comparisons for flexure and tension test methods, in addition to the intercomparison. The materials to be tested will be mainly unidirectionally reinforced epoxy matrix systems using either glass fibres or carbon fibres. Depending on material availability tests will also be undertaken on carbon fibre/PEEK and aramid fibre/epoxy systems.

To date 24 participants have registered for the overall programme. This includes France (3), Italy (4), Japan (3), Portugal (1), United Kingdom (12) and West Germany (1). The participants include industry, universities and research laboratories, both government and non-government. In recognition of the time consuming nature of fatigue testing and the difficulty of obtaining sufficient machine-time all of 1990 has been allocated for testing. Results are due at NPL in January 1991.

The background and development of the round robin programme are discussed in "A VAMAS round robin on fatigue test methods for polymer matrix composites. Part 1. Tensile and flexural test methods of unidirectional material" by Dr Graham D Sims. NPL Report DMA(A)180 (1989).

Technical Working Area 6

SUPERCONDUCTING AND CRYOGENIC STRUCTURAL MATERIALS

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The 5th TWA meeting was held on 24 July at the 1989 International Cryogenic Materials Conference (ICMC) at UCLA, California, USA. Twenty seven participants from eight countries attended the meeting where detailed results, new proposals and future schedules were discussed.

The first intercomparison on critical current (I_c) measurement using three Nb_3Sn wires and different measurement techniques has been completed. The second intercomparison using one common Nb_3Sn wire and the same specimen holder was approved at the meeting, and the test guideline will be distributed shortly. A questionnaire on test facilities capable of I_c measurement for large scale superconductors will also be distributed at the beginning of next year.

The results of the intercomparison on AC loss measurement in Nb-Ti wires are being analysed. The coefficient of scatter in AC loss was about 10% which was rather small considering the variety of measuring methods. A comprehensive AC loss measurement with more strictly defined conditions is under consideration.

In the area of cryogenic structural materials, results of tensile and fracture toughness round robin tests at 4.2K on SUS316LN and YUS170 steels were summarised and analysed. Another round robin tensile test using new alloy materials with attention to load cell calibration, microstrain measurement and serration study is being planned. Another fracture toughness round robin test with attention to strain rate, size effects and crack growth effects will also be included. A new programme on strain gauge calibration at cryogenic temperatures is proceeding.

Meanwhile, at the 1989 ICMC a VAMAS session was established, where in total, 10 related papers were presented. The time and location of the next TWA meeting in this area will be fixed shortly.

Technical Working Area 7

BIOENGINEERING MATERIALS

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Representatives of international groups in the VAMAS activity have met recently and are now proposing a new VAMAS round robin on cell compatibility tests for a number of potential advanced bioengineering materials. Cell compatibility tests generate useful results quickly enabling quantitative evaluation of toxicity to be made. However, the test methods are different in each laboratory with many variations in test procedure including cell types, media, physical form of test specimen and the manner of contact between specimens and cells. All these different experimental factors can influence the final results of the method and so inhibit comparisons of data from different laboratories. The objective of this VAMAS TWA round robin thus would be to produce the data necessary for establishing standard cell compatibility tests. In the round robin, mouse fibrobrast cells (L929) would be cultivated on disk specimens of four candidate materials, alumina, hydroxyapatite, zirconia and Ti-6Al-4V. The precise details of the procedure to be tested have been proposed and specimens from Japan would be sent to participating groups upon acceptance of this proposal by the Steering Committee.

Technical Working Area 8

Hot Salt Corrosion Resistance

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Following the preparation of the guideline document on hot salt corrosion testing, which has been widely circulated, final arrangements have been made for the international intercomparison to investigate the validity of the procedures described in the guidelines. Testpieces have recently been supplied to all potential participants in the expectation that corrosion testing can take place during the next six months. Several Japanese organisations have already conducted corrosion testing in accordance with the guideline document and the results are being evaluated.

In view of the widespread interest in improving measurement and analysis techniques for hot salt corrosion resistance, a special edition of the journal "High Temperature Technology" has been produced. This edition will describe the state-of-the-art in this important area of materials technology and will focus specifically on the work of VAMAS in improving measurement practice.

WELD CHARACTERISTICS

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The investigation of weld penetration as a function of welding parameters and trace element content has been completed for two types of austenitic steel. The results are now being evaluated and a final report will be produced. Initial indications suggest that the sulphur content affects the weld profile during Tungsten Inert Gas (TIG) welding in these materials and the results are broadly consistent with the predictions of surface-tension driven flow. However, the variations in profile were not large in some cases due in part to the relatively small differences in sulphur content from cast to cast. Differences in welding procedures may also have influenced the results.

Overall the results appear to support the need to specify a minimum level for certain surface-active elements in order to ensure good weld quality in TIG welding.

Technical Working Area 10

MATERIALS DATABANKS

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TWA 10 examines issues related to developing standards for building and using materials databases. Progress has so far been achieved through the following activities:

Phase 1: Consensus Report of a VAMAS Working Group:
FACTUAL MATERIALS DATABANKS -
THE NEED FOR STANDARDS
VAMAS Technical Report No 2, July 1987.

Phase 2: Prestandardisation Projects:
- Interlaboratory Comparison of Data Evaluation Methods, NRIM, May 1988
- VAMAS Workshop on Standards for Materials Databanks, Petten, Nov. 1988
- Inventory of Materials Designation Systems, K W Reynard, UK, May 1989

The Phase 2 projects have reached an advanced or concluding status, and some have resulted in proposals for follow-up activities in a third phase.

Interlaboratory Comparison of Data Evaluation Methods

This round robin, in which 14 institutions are participating under the leadership of Dr S Nishijima of the National Research Institute for Metals (NRIM) in Tokyo, is nearing completion. It has already made available data from creep rupture, creep strain-time, HCF and fatigue crack growth tests. In testing a set of models and their numerical procedures currently used for the evaluation of materials properties in databases, the participants encountered various expected and unexpected problems by which the workability and the results of the methods are impaired or made unreliable and incomparable. Most of the problems are linked with the lack of harmonised standards and protocols, demonstrating the need for better standards, not only for database access, data, terminology and designations but also for methods of evaluation and analysis. The final report will become available at the beginning of 1990.

VAMAS Workshop on Standards for Materials Databanks

An extensive summary of this workshop (JRC Petten, November 1988) was presented in Bulletin No 10. Reports have been published in ASTM Standardisation News, June 1989 (J Rumble: Making Materials Database Standards International - The Next Steps) and as VAMAS Report No 4 (P Büttner and H Kröckel (eds): Report of a VAMAS Workshop on Standards for Materials Databanks, November 1989).

As a follow-up to the conclusions of the "Data Interchange" group of this meeting, a special Materials Data Interchange Format (MDIF) Workshop attended by 30 experts was organised in September 1989 at Derby, UK, hosted by Rolls-Royce. The Workshop has identified MDIF as a top priority to support exchanged data between materials databases and facilitate reuse of data entry files. The participating organisations from Europe and the USA agreed to test two pragmatic data interchange methods, the Cambridge Tabular Data Interchange Format (CTDIF) presented by P M Sargent, and a physical file format based on work in PDES/STEP, the American and ISO standardisation activities for product data exchange.

Inventory of Materials Designation Systems

This activity, led by K W Reynard, UK, aims to draw attention to the existing discrepancies and the confusion between standards, classifications and designation systems. This work is an extremely important first step to the development of a harmonised materials designation system(s) suitable for computer databases. The word "Systems(s)" is written in the plural because work has shown that existing systems are very complex, use different technical factors as their basis, and will probably not be accommodated by a single all encompassing system. A preliminary report is being drafted that will be circulated to standards bodies throughout the world for comment. The report will also pave the way for cooperative action.

Proposals Under Discussion for Phase 3 Activities

- Constructing an Inventory of Methods/Models for Data Analysis/Evaluation: This inventory or catalogue should describe the methods, input and resulting data as a step towards harmonisation and standardisation of the methods.
- Inter-institution Comparison of Materials Data Interchange Protocols: This round-robin proposed by the Derby Materials Interchange Workshop is a candidate activity for coordination by TWA 10, and will be submitted to the VAMAS SC for approval.

Technical Working Area 12

EFFICIENT TEST PROCEDURES FOR POLYMER PROPERTIES

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Four of the five participating nations (UK, USA, FR Germany and Japan, but not France) have conducted assessments of their country's uses of accelerated durability testing (ADT), identifying preferred tests, their principal features, the reasons for their use, the need for improvements and the potential for harmonisation. Studies of the status of analytical methods for predicting long term performance from data from short term accelerated tests have not been completed yet. All aspects of the work of TWA 12 were discussed at a meeting in Paris in September 1989, and decisions on further work were made. It was agreed that TWA 12 would concentrate its immediate activities on two topics:

(a) preparation of a concise general guide to the use of accelerated durability tests (to be edited by R Brown, UK),

(b) a study of the potential of standard reference materials for ADT (to be led by G Zerlaut, USA). TWA 12 will re-establish and maintain contact with ISO/TC 61/SC 6.

Further meetings of TWA 12 are planned for March/April 1990 in London and September 1990 in Florida.

Technical Working Area 13

LOW CYCLE FATIGUE

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This intercomparison programme of high temperature, low cycle fatigue tests is aimed at examining the effects of testing variables and material characteristics.

The programme involves testing four materials, classified according to their cyclic strain characteristics as strain-hardening (AISI 316L steel), strain-softening (9Cr1Mo steel and IN718 alloy) and strain-stable (Nimonic 101 alloy).

The initial findings, based on a preliminary evaluation of the results available to September 1989, indicate that:-

(i) The variation in mean lives between laboratories is, in general, significantly greater than that of results within an individual laboratory.

(ii) The extent of both scatter in individual results and interlaboratory variation appeared to be influenced by material characteristics.

(iii) The variation in stress range at half-life at a given strain range was generally 3 to 4 times greater when considering the results from all the laboratories than that observed within any one laboratory.

More detailed examination of these results, together with others now received, is expected to indicate the means to improve interlaboratory reproducibility of high temperature LCF data.

THE TECHNICAL BASIS FOR A UNIFIED CLASSIFICATION SYSTEM FOR ADVANCED CERAMICS

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This activity has three prime objectives:

1. To identify and assess the issues inherent in developing a classification system for advanced ceramics.
2. To establish a building-block structure necessary for international use.
3. To develop mechanisms and institutional links for system implementation.

The membership of the Technical Working Group has been expanded to sixteen for greater industrial representation of the advanced ceramics community. Additional members are anticipated so that the final makeup of the Working Group will include at least two from each VAMAS member nation, representing the interests of industry, academia and standards organisations.

Activities for this reporting period are related primarily to data gathering and follow-on actions resulting from the first meeting of the Working Group, held on April 6-7, 1989 at Bundesanstalt für Materialforschung und-prüfung (BAM) in West Berlin to map out strategy and set plans for project implementation. Items of work in progress include a review of terminology needs for advanced ceramic classification; the development and issuance of a survey questionnaire on advanced ceramics classification needs, scope and basis; the organisation of a general interest classification workshop and the development of a worldwide list of "who's who" in advanced ceramics and standards. The following summarises major progress to date:

1 A tentative "working definition" of advanced ceramics was formulated to set the range of products to be covered by the classification system. A broad scope was adopted and includes all major functional types, ie structural, electronic, magnetic, etc. This definition will be revised as more information is obtained, but current views point to the direction of a definition that will allow incorporation of product types called "engineering ceramics" and "technical ceramics" as these are fore-runners of the more popular term "advanced ceramics".

2 A classification questionnaire, originally prepared and distributed in the US by the United States Advanced Ceramics Association (USACA), was revised to fit other VAMAS nations customs and needs. Distribution to select organisations in the CEC countries, Canada and Japan is underway with responses from France, Italy and the UK in hand. It is anticipated that a preliminary analysis of the survey will be completed by January 1990.

3 Plans for a classification workshop, scheduled for June 21-22, 1990 at the Joint Research Centre-Ispra, Italy, (alternate location JRC-Petten) are being finalised. This will precede an associated conference, the 7th CIMTEC World Ceramics Congress, Montecatini Terme, Italy, June 24-30, 1990, at which an update of

the VAMAS classification project will be presented in a special advanced ceramics standards session.

4 An advanced ceramics contact list has been compiled and includes over 1200 entries covering the US, the CEC countries, Canada and elsewhere. The list can be made available to interested persons.

5 A meeting of the Working Group was held at the National Physical Laboratory, Teddington, England, December 11-12, 1989 and will be reported on in the next issue of the VAMAS Bulletin. The working group encourages participation by others at future meetings.

Contact S J Schneider, NIST, for further information on the VAMAS project on the classification of advanced ceramics.

VAMAS CALENDAR

Polymer Blends National Technical Working Area meetings:

Japan, France	November 1989
Italy	December 1989
UK	January 1990

Advanced Ceramics Classification Working Group meeting:

NPL, UK	December 11-12 1989

Creep Crack Growth TWA,

NPL, UK	March 29-30 1990

Sixth International meeting of Polymer Blends TWA (Immediately after Polymer Processing Society meeting on April 17-20):

Nice, France	April 21-22 1990

VAMAS Steering Committee meeting:

Paris, France	April 26-27 1990

Advanced Ceramics Classification Workshop:

JRC, Ispra, Italy	June 21-22 1990

3rd European Symposium on Polymer Blends (PRI), Robinson College, Cambridge University, including Polymer Blend TWA International Meeting on Mechanical Testing (organised by Dr I K Partridge):

UK	July 24-26 1990

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