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

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Cover: Scanning electron micrograph of Lichtenberg figures (treeing) due to surface breakdown of a polycrystalline yttria specimen bombarded with 40 kV electrons and subsequently discharged with low electrons; from project 11 in TWA 2 illustrating how the investigation of charging phenomena in insulators is helping to explain the mechanism of dielectric breakdown and fracture.

Photograph by courtesy of Dr Claude Le Gressus, Centre D'Etudes Nucleaires de Saclay, France



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Feature Article

VARIABLE PENETRATION IN WELDING

A VAMAS COOPERATIVE PROJECT by K C MILLS AND T B GIBBONS Division of Materials Metrology National Physical Laboratory Teddington, Middlesex, UK, TW11 0LW

Introduction

In the automatic welding of steel using the Tungsten Inert Gas (TIG) process the degree of weld penetration can vary for different batches of steel of nominally the same composition. Weld penetration depends on the fluid dynamics in the molten pool which can be influenced by (i) the electromagnetic forces, (ii) the aerodynamic drag at the surface, (iii) buoyancy forces in the melt and (iv) surface tension driven or Marangoni flow. The observation that similar variability in weld penetration can also occur during laser or electronbeam welding has focussed attention on surface tension driven flow as the major factor influencing the weld profile.

It has been established that poor weld penetration in steels is usually associated with very low levels of sulphur ie <50 ppm and that satisfactory welding can usually be achieved with sulphur levels >100 ppm. Heiple and Roper⁽¹⁾ developed a simple model based on the temperature sensitivity of surface tension and the implications for weld pool fluid dynamics to describe the effect of surface tension driven flow on weld profile. The principles of the model are summarised in Figure 1.



Figure 1. Schematic diagram illustrating the Heiple-Roper model.

In circumstances where the temperature coefficient $\left(\frac{d\gamma}{dt}\right)$ of surface tension is positive the temperature distribution at the surface is such that an inward and downward fluid

flow occurs. This gives a satisfactory weld profile but when the value of $\left(\frac{d\gamma}{dr}\right)$ is

negative the fluid flow along the surface is outward from the centre and poor weld penetration results. Measurement of the surface tension of ferritic and austenitic steel has confirmed that when the concentration of surface active elements, eg sulphur,

was low, surface tension values were high (>1700) mNm⁻¹ and $\left(\frac{d\gamma}{dt}\right)$ was negative.

The reverse was true for higher concentrations of surface active elements. Thus the Heiple-Roper model provided a self-consistent explanation for the variability in weld penetration observed in practice although the approach was novel and did not meet with universal acceptance within the welding fraternity.

The purpose of the VAMAS project was to carry out a systematic study of weld profiles for steels with different levels of surface-active elements in order to assess the validity of the Heiple-Roper model and to provide a basis for the specification of residual elements in steels to ensure consistent weld profiles.

Summary of Results

The materials used were the austenitic steels of types 304 and 316 supplied as sheet ~ 3 mm and ~ 2 mm thick respectively and each was available with two levels of sulphur as shown in Table 1. The participants in the project are shown in Table 2 and procedures for carrying out the welding trials and for measuring weld penetration were agreed at the outset. Two methods were used to measure weld profiles. For partial penetration the depth to width ratio d/W was determined and for full penetration welds the ratio of the back face width to the front face width (W_b / W_f) was used, Figure 2.

A key factor in correlating weld characteristics with surface-tension driven flow is the ability to make accurate measurements of surface tension as a function of temperature for samples of all the steels. The technique developed at the National Physical Laboratory uses the levitated drop principle to give highly accurate measures of surface tension at temperatures up to 1800 °C and measurements were made using this method on both batches of each steel.







Full penetration weld

Figure 2. Schematic representations of weld profiles.

Table 1										
Chemical Analysis of Steels										
Mass %										
Steel	С	Si	Mn	Р	S	Cr	Ni	Мо		
304 LS	0.06	0.57	1.06	0.032	0.003	18.09	8.45	-		
304 HS	0.06	0 .41	0.86	0.024	0.008	18.29	8.4	-		
316 LS	0.05	0.54	0.84	0.024	0.001	17.58	11.74	2.07		
316 HS	0.06	0.47	0.82	0.028	0.005	17.55	11.83	2.25		

Table 2						
Participants	Establishment	Work carried out				
Dr H Nakamura Dr A Okada	National Research Institute for Metals (NRIM), Japan	Welding trials				
Prof K Ishizaki Prof S Yokoya Dr T Okada	Nippon Institute of Technology (NIT), Japan	Welding trials Simulation experiments				
Prof A Matsunawa et al	Welding Research Institute Osaka University, Japan	Mathematical model Simulation experiments				
Prof D Aidun	Clarkson University, USA	Welding trials				
Prof T Eager Dr M A Kahn	MIT, USA	Welding trials				
Dr P W Fuerschbach Dr J L Jellison	Sandia National Laboratory (SNL), USA	Welding trials				
Dr K C Mills Mr R F Brooks	National Physical Laboratory (NPL), UK	Surface tension measurements				
Mr D Harvey	Welding Institute (WI), UK	Welding trials				
Mr A Shirali	Strathclyde University, UK	Welding trials				
Mr O Cadman	GEC Turbine Generators, UK	Welding trials				
Dr J Lambert	CEGB (now PowerGen), Marchwood Laboratory, UK	Welding trials Statistical analysis				
Dr J Binard	Excel 'S', France	Welding trials				
Dr G Tsotridis	Petten Establishment, JRC	Mathematical model				

The results of the partial weld penetration studies are shown in Figure 3 where the ratio of d/W is plotted as a function of lineal energy. E, for both casts of each steel. The lineal energy is a parameter that enables comparison to be made for different welding conditions and is defined as

$$E = \frac{VI}{L}$$

where V is the welding potential, I is the current and L the welding speed.

It is clear from Figure 3 that, although there is some variability in the data, there is a general trend to deeper weld profiles for the higher sulphur materials ie a result broadly in line with the Heiple-Roper model. The results for the full weld penetration showed little evidence of a correlation and it was concluded that in these thin sheet materials the ratio Wb /Wf was a poor measure of weld penetration.

Results of surface tension measurements for the steels are shown in Figure 4 and it can be seen that in each case the high sulphur content corresponds with lower values of γ and a positive temperature coefficient. However it is evident that the effect is not particularly strong in the case of the 316 steel and this is consistent with the sulphur levels in this material. It has been estimated⁽²⁾ that sulphur contents of 80 ppm are required to ensure a positive temperature coefficient for surface tension



(a)

(b)





Figure 4 - Surface tension results for the high (o) and low (x) sulphur contents of 304 (a) and 316 (b) stainless steels

measurements so that on this basis the "high" sulphur level in the type 316 steel was somewhat marginal.

Two mathematical models^(3,4) were developed to predict the influence of the various factors that control weld-pool fluid dynamics on the weld profile. It was shown that Marangoni forces were predominant in normal welding conditions⁽³⁾ but that aerodynamic drag becomes significant when long arc lengths are used⁽⁴⁾.

Conclusion

It has been shown that for TIG welding carried out under different conditions the weld profiles obtained were broadly consistent with the Heiple-Roper model for the effect of surface tension driven flow on fluid flow in the weld pool. The results suggest that control of surface active elements, particularly sulphur, could have a marked effect on weld profile and consequently where low levels are not required from toughness considerations a lower limit for sulphur should be specified. Elements such as calcium and zirconium which have a strong affinity for sulphur should also be controlled if appropriate fluid flow conditions are to be achieved in the weld pool.

With the completion of this project no further work on this topic is planned within VAMAS. However it appears likely that further progress towards setting specification limits for surface-active elements in steels may be made through the International Institute of Welding.

References

- 1. HEIPLE, C R and ROPER, J R. Welding Journal. 1982, (4), 72.
- MILLS, K C, KEENE, B J, BROOKS, R F and OLUSANYA, A. The surface tensions of 304-type and 316-type stainless steels and their effect on weld penetration. *Proceedings of the Centenary Conference of the Metallurgy Department*, Strathclyde University, Glasgow, June 1984, edited by H B Bell and D B Downie, 1984.
- 3. TSOTRIDIS, G, ROTHER, H and HONDROS, E D, Naturwissenschaften. 1989, (76), 216.
- MATSUNAWA, A, YOKOYA, S, OKADA, T and ASAKO, Y. Model experiments of weld pool convection and penetration shape in TIG arc welding. *Paper submitted at meeting* of the VAMAS project on Variable Weld Penetration, National Physical Laboratory, Teddington, UK, December 1988.

Technical Working Areas

INTRODUCTION

The Memorandum of Understanding, under which VAMAS operates, runs for five years from June 1987 - 1992. Now is a good time to take stock and examine how each TWA is succeeding in meeting its broad objectives. Also, during the remainder of 1990, the VAMAS Steering Committee will be developing a case for a second phase of VAMAS to start from mid-1992 when the present Memorandum of Understanding comes up for review. An important part of this process is an assessment of the progress made by each TWA. Consequently in this issue of the Bulletin, the regular reports from the TWAs have been replaced by brief precis of their respective objectives and achievements.

Technical Working Area 1 WEAR TEST METHODS

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OBJECTIVES

- Improvements in the reproducibility and comparability of wear test methods by developing internationally agreed wear test methodologies.
- Characterisation of the wear behaviour of "advanced" materials in comparison with conventional materials, advanced materials being defined as ceramics and inorganic coatings.

- Performance of two round robin interlaboratory comparisons with 32 (first round) and 38 (second round) laboratories from all member countries of the Economic Summits plus Denmark and Finland; investigation of the tribological behaviour of advanced ceramic materials (alumina, silicon nitride and AISI 52100 steel) under dry sliding conditions.
- Evaluation of the experimental results and publication of these in: Wear (1987, 1989), VAMAS Bulletin (1987, 1988), Tribology (1988), NIST-IR (1989).
- Preparation of VAMAS Technical Note 1 providing a methodology for improvements in the reproducibility and comparability of wear tests (VAMAS Bulletin 7, January 1988); the VAMAS Technical Note has been forwarded to the national and international standardisation bodies; it was adopted by ASTM and DIN.
- Survey on future requirements for wear test methods indicating the necessity to continue work with respect to
 - (i) hard coatings
 - (ii) specimen geometry
 - (iii) variation of testing conditions (in particular load and speed).

Technical Working Area 2 SURFACE CHEMICAL ANALYSIS

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OBJECTIVES

- The overall objective of TWA 2 is to ensure that data reported using surface chemical analysis techniques may be provided with confidence and certainty. An initial subset of techniques to be examined were Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectroscopy (SIMS), together with sputter-depth profiling (SDP). As projects evolve and cross-calibrations are established, other techniques, such as nuclear reaction analysis, Rutherford back-scattering spectrometry, etc, will be included.
- Five major aspects have been identified:
 - (a) an agreed base of principles, definitions and equations for the relevant aspects of the techniques
 - (b) reference procedures specifying how measurements should be made,
 - (c) reference procedures, material and data to characterise and calibrate all instrument parameters
 - (d) reference data of relevant parameters to insert into the appropriate equations
 - (e) the infrastructure necessary for setting standard methods of specifying an analysis.

- Over the period to date the SCA Programme has developed 25 projects with leaders in all Member States. Over 100 laboratories have been involved in these projects and about 15 papers have been presented at scientific meetings where the results of VAMAS coordinated activities have been highlighted. In this way this TWA has considerably raised the public's awareness of the issues to be tackled and the methods to be adopted to solve problems in this broad area of enabling technology. The coordinated activity has led to greater efficiency and acceptability of the resulting research.
- Three of the projects have been completed, (a), (b), and (c) below, and these in turn have catalysed further work
 - (a) The first of the projects, Project 1, concerned the development of a reference material of tantalum pentoxide on tantalum to be used to calibrate sputtering rates and interface resolution in SDP. It is difficult to measure the ion beam current densities in commercial surface analysis

systems and work on sputtering shows that the Faraday cups typically used involve considerable systematic errors. These errors may be diagnosed and calibrated using the reference material. In this way flux density measurements were improved to a level appropriate for present and foreseeable work. The material also allows calibration of the sputtering uniformity in the ion beam

- (b) The second project, Project 16, used the above material to show that the non-destructive angle-resolved XPS and the SDP methods can be used to give the same thicknesses for a carefully designed thin oxide layer reference material. This gives credibility to ARXPS for the non-destructive quantitative assessment of ultra-thin films
- (c) The most recent project to be completed, Project 10, concerns the need to transfer data files from laboratory to laboratory and, to this end, a Standard Data Transfer Format has been established, published and accepted in all Member States.
- Further projects are close to finishing and involve the energy and intensity calibrations of Auger electron spectrometers. These two scales define the spectral result. By the use of reference materials, calibration methodologies have been proven which establish these at ±0.05 eV and ±2 %, respectively. Both of these levels are sufficient for present work and represent over an order of magnitude improvement on the prior situation.

Technical Working Area 3 CERAMICS

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OBJECTIVE

 To undertake research investigations into the reliability and reproducibility of test procedures for advanced technical ceramics prior to formal standardisation.

- An interlaboratory round robin has been conducted to ascertain the ability of laboratories to undertake "dynamic fatigue" tests (tests at different loading rates) to measure the subcritical crack growth rate exponent. The test reproducibility was first determined by NIST over about six orders of magnitude in stressing rate. Participating laboratories demonstrated that useful results could only be obtained if at least three orders of magnitude in rate were used. Very poor results were obtained if two or less orders of magnitude were used. This result is of importance in setting the conditions in formally standardised test procedures, and is now being considered in ASTM C28 and CEN TC184 standardisation committees.
- An interlaboratory round robin organised by NPL has been conducted to examine the reproducibility of hardness measurements on two ceramic materials of fine and coarse-grained microstructures. The results have shown that with existing techniques for metallic materials very considerable errors can arise, especially with microhardness measurements, to the extent that hardness is not a very good characteristic to use in materials specifications. Guidelines on the use of hardness measurements are being prepared for consideration by standards committees.
- The Japan Fine Ceramics Centre has organised a round robin exercise on fracture toughness test procedures. Three procedures have been examined using single sets of test bars of two materials, a sintered silicon nitride and a zirconia/alumina composite. The results are still being evaluated, but preliminary assessment has shown that there are possibilities for considerable scatter between laboratories if test procedures are not clearly followed. In addition, the different tests gave significantly different results, which may be attributed in part to the difference between short crack and long crack fracture toughnesses. Further work is required in this area.

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. No standard test procedures specific to these materials have yet been published.

Polymer blends are thermodynamically immiscible mixtures of two or more polymers. Properties are structure dependent arising from morphological variability introduced during compounding and forming. In most cases the specimens are anisotropic with the morphology and stress field frequently changing during the tests. Optimisation of blend performance usually requires addition of an ingredient responsible for modification of the interface, the compatibiliser. Thus, owing to variability of blend interphase properties and morphology, the use of existing standard tests (developed for single-phase polymers) is rarely possible.

In general, the standard test procedures can be divided into two groups: those dealing with materials and others with finished products. In both cases either a single point or functional information is being sought. The TWA aims to provide data for functional characterisation of raw materials, the commercial polymer blends. Furthermore there are three types of polymer with widely different solid state behaviour; rubbery (R), glassy (G) and semi-crystalline (C). Because in blends one polymer provides a matrix in which the other is dispersed, the behaviour of blends will depend on the distribution and size of R, G and C. To be certain that proposed test methods are of general applicability requires the examination of several blend types.

- Currently the specific objective of the TWA is to examine test procedures and provide data on raw materials and materials which have been processed in a well characterised way. Because standard test procedures are non-existent, the work of the TWA is being carried out in five parallel and mutually supportive technical areas
 - (a) Melt flow
 - (b) Dynamic testing
 - (c) Thermal properties
 - (d) Morphology
 - (e) Mechanical properties.

The programme has been conducted in two phases, I and II. Phase I examined the specially made, highly immiscible blends of polycarbonate with polyethylene (PC/PE). Phase II makes use of commercial blends. The primary aim of Phase II is to examine applicability of test procedures identified in Phase I and to develop the technical basis for their adoption by the Standardising Organisations. The first of the commercial blends examined in Phase II was ORGALLOY R-6000. It is expected, because of the diversity of morphological forms, anisotropy in structure and polydispersity of domain sizes, that items (d), and (e), will present a challenging task.

- In Phase I four tons of material was distributed to about 100 laboratories. Results from work in Phase I have resulted in a proposed standard test method "Thermal Analysis of Immiscible Polymer Alloys and Blends" which is being examined as a new work item T550 by ISO TC 61. Also several test methods were selected for further examination in Phase II.
- The TWA has significantly contributed to the scientific understanding of polymer blends; 15 articles in scientific books and journals, 17 articles in conference proceedings and four reports have been published. International collaboration has been enhanced. Extensive round robin tests have enabled instruments and test procedures in laboratories around the world to be validated. Incorporation of test procedures recommended by the TWA provides immediate savings to the collaborating laboratories.
- The first results from Phase II on ORGALLOY were presented during the 6th Annual meeting of the TWA in Nice, April 1990. The thermal properties, measured according to the method developed in Phase I, established melting temperatures to ±1 °C and enthalpies of fusion to ±0.5 %. The dynamic test methods in solid and molten state are well advanced. It is expected that shortly two new work items based on this work will be transferred for examination by ISO TC 61.
- A need for the development of qualitative analysis methods for describing morphologies of immiscible polymer blends has been identified. This information is essential for the quantitative interpretation of any polymer blend data, particularly that obtained in the solid state.
- Work on mechanical properties has highlighted the need for careful evaluation of structure - property relationships in polymer blends in order to improve the reliability and predictive capabilities of future test methods.

Technical Working Area 5 POLYMER COMPOSITES

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OBJECTIVES

- To assess and refine the measurement of fracture toughness for delamination crack growth using Mode I (crack opening) and Mode II (shear) test conditions.
- 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.

ACHIEVEMENTS

Delamination Test Methods

- Phase I round robin tests have been completed. The programme extended an ASTM round robin programme to a wider range of materials (continuous aligned and fabric glass and carbon fibre systems) and to both thin and thick sections. The programme involved 14 laboratories. The results were collated in the annual report of the working group "Delamination tests of DCB and ENF composite specimens in Mode I and Mode II" R Khoshravan and C Bathias, April 1990. The results were also discussed at a joint meeting held in Orlando (Nov 1989) with representatives from ASTM, JIS and EGF (European Group on Fracture). The meeting agreed to produce a joint international protocol for these test methods. The programme and protocol have been prepared by the EGF and involve 35 laboratories world wide. Results are due back by September 1990.
- A second phase of the programme is currently under way using fatigue crack growth conditions. A similar range of materials and test geometries as examined in Phase I are in use. Results are due in September 1990.

Fatigue Test Methods

 An agreement has been reached on the parameters that will be examined. Both flexure and tensile testing of unidirectionally reinforced materials will be studied. Laboratories from the United Kingdom, France, West Germany, Japan, USA, Canada and Italy will participate. Results are expected by the end of 1990.

Creep Test Methods

 A creep round robin led by Japan, and organised by the French VAMAS group, is shortly to commence.

Technical Working Area 6

SUPERCONDUCTING AND CRYOGENIC STRUCTURAL MATERIALS

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OBJECTIVES

- The establishment of reliable measurement techniques for superconducting and cryogenic structural materials leading to eventual standardisation.
- To conduct round robin tests on critical current measurements, I_c, in Nb₃Sn multifilamentary wires which are capable of generating magnetic fields over 10 T. I_c is the most important superconducting property for practical application.
- To conduct round robin tests on AC loss measurement in Nb-Ti multifilamentary wires. The development of superconducting wires for AC use is an urgent up-to-date subject.
- To conduct round robin tests on tensile and fracture toughness measurements at 4.2 K in cryogenic structural materials. These tests are indispensable for the construction of reliable superconducting equipment. A round robin test on strain gauges at cryogenic temperatures is also being performed.

- The round robin test on I_c measurement in Nb₃Sn wires with 24 participant laboratories from 9 different countries was completed in 1988. The strain caused by the difference in thermal expansion coefficient between Nb₃Sn and the measurement mandrel was found to be the main origin of scatter in I_c. The second round robin test under more specified conditions using the same measurement mandrel has been recently started to define recommendations for standardisation, and will be completed by the end of 1991.
- The round robin test on AC loss measurement using four different Nb-Ti wires was completed in 1989. The results are being analysed, and complementary tests will be performed soon. The recommendations for AC loss measurement will be summarised at the end of 1991.
- The round robin test on tensile measurement at 4.2 K on 316LN and YUS170 steels was completed in 1988. The data were reasonably consistent. However, strain measurement caused an appreciable scatter in data. Thus, a round robin test on strain gauges at cryogenic temperatures was initiated in 1989. 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. From this year the second round robin tests on tensile and fracture toughness

measurements at 4.2 K using a superalloy and a Ti alloy under more refined conditions have been started, and will be completed at the end of 1991.

 In total 20 papers have been published so far in technical journals and proceedings resulting from VAMAS cooperation in this TWA. If the VAMAS cooperation is renewed from 1992, it will be a good time to start the standardisation of measurement techniques for new high-Tc superconductors and composite cryogenic structural materials.

Technical Working Area 7 BIOENGINEERING MATERIALS

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OBJECTIVES

To conduct an international pre-standardisation round robin on the biocompatibility of bioengineering materials, specifically to produce data necessary for establishing widely agreed cell compatibility tests. The round robin will examine the effects of variations in test procedure such as for example cell types, media, specimen form and cell/specimen contact. In this initial work mouse fibroblast cells will be cultivated on disc specimens of the following materials: hydroxyapatite, alumina, zirconia, Ti-6AI-4V and UHMWPE.

- Preliminary tests have been performed in Japan to define in detail the parameters which the round robin will examine.
- Groups from USA, UK, France, Italy and Japan have already agreed to take part.

Technical Working Area 8 HOT SALT CORROSION RESISTANCE

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OBJECTIVE

 The purpose of the programme is to develop a unified procedure for assessing the hot salt corrosion resistance of superalloys in burner-rig tests.

ACHIEVEMENTS

- A guideline document has been produced for burner-rig testing based on the concept of contaminant flux rate as the unifying parameter for consistent and inter-comparable data.
- Arrangements have been made for a new international intercomparison to probe the validity of the testing guidelines.
- A procedure for statistical assessment of corrosion attack has been established.

The project is currently at the stage where materials have been distributed to allow the intercomparison to go ahead. Both coated and uncoated materials are involved and test conditions have been chosen to give reasonable levels of attack in the 500 hour duration of the test. Laboratories in USA, Europe and Japan are taking part and results should begin to become available within the next six months.

Technical Working Area 10 MATERIALS DATABANKS

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OBJECTIVES

- VAMAS Technical Working Area 10, Materials Databanks, works on issues related to the developing standardisation which significantly impacts the building and use of computerised materials databases. This working area has the primary objectives to:
 - (i) assess the role that standards can have in the computerised flow of materials information
 - (ii) identify needs, particular problem areas and options for standardisation activity
 - (iii) define, organise and coordinate pre-standardisation research which addresses projects of international scope and transborder impact.
- TWA 10's objectives are correlated with the activities of other international, national and regional institutions, which are involved in the computerisation of materials information and related enabling and standardising activities, in particular CODATA, the CEC, ISO, ASTM, CEN-CENELEC. TWA 10 endeavours to supplement and support the objectives, activities and impact of these institutions in the field of materials data systems where it offers the benefits of a world wide dimension.

ACHIEVEMENTS

- Progress in TWA 10 activities can at present be described in three phases: a first survey oriented phase which was concluded in 1987, a second phase of preparatory and pre-standardisation activities that is near to conclusion, and a third phase in which new pre-standardisation projects are planned.
- 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

- VAMAS Workshop on Standards for Materials Databanks, JRC-Petten, November 1988 (VAMAS Technical Report No 4, November 1989). - International Workshop on Materials Data Interchange, Rolls-Royce plc, Derby, September 1989 (Follow-up meeting of the Data Interchange Group of the VAMAS Workshop at Petten).

- Inventory of Materials Designation Systems, characterising features and structures of all known materials designation systems world-wide. Report by K W Reynard, UK, well advanced.

- Interlaboratory Comparison of Data Evaluation Methods, organised and evaluated by NRIM, Tokyo. 14 participating institutions; final report (in two parts) by S Nishijima, Y Monma and K Kanazawa (NRIM), to be published shortly.

Phase 3

- Projects recently approved for start in 1990:
- Inventory of Methods/Models for Materials Data Analysis and Evaluations.
- Inter-institution Comparison of Materials Data Interchange Protocols.

Technical Working Area 11 CREEP CRACK GROWTH

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OBJECTIVE

 The overall aim is to develop a unified approach to the measurement and interpretation of crack growth data.

ACHIEVEMENTS

- The results of three separate intercomparisons of crack growth in a CrMoV ferritic steel have been analysed using an agreed procedure and have shown an excellent level of agreement for the steady-state regime of crack growth.
- A procedure has been devised for interchange of data among participants.
- Agreement has been reached on the relevance of the various crack-tip correlating parameters in terms of the mechanics of deformation and fracture.
- Validity criteria have been established for crack growth measurements.

At the completion of Phase I, procedures had been agreed for measurement and interpretation of steady-state crack growth in materials with reasonable levels of creep ductility ie ~15 %. However difficulties remain with interpretation and modelling of the early stages of crack growth and this is the main area of activity in Phase II. The aim is to examine some new approaches to the modelling of initial crack growth and to interpret the results in the context of data already available from the intercomparisons.

A draft document, which should in due course become a standard relevant to steady-state crack growth, has been produced in collaboration with ASTM and is now subject to further scrutiny and evaluation.

Technical Working Area 12

EFFICIENT TEST PROCEDURES FOR POLYMER PROPERTIES

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OBJECTIVES

- To characterise the long-term properties of the time-dependent viscoelastic materials. Relevant properties include creep, stress relaxation, fatigue and durability in aggressive environments. In order to avoid prohibitive amounts of testing over long timescales, it is common practice to make use of extrapolation, interpolation, inter-conversion of property data or accelerated tests. Many of these 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 water, with particular reference to accelerated tests. The broad objectives of the work are to provide support to standardisation activities in this field, with particular attention to the interpretation of data (ie deduction of long-term performance from short-term accelerated tests).
- Current work is addressing two topics:
 - (1) provision of a general guide to the use of accelerated durability tests (ADT), and is being coordinated by R Brown, RAPRA Technology Ltd, Shrewsbury, UK, SY4 4NR and could, in due course, provide the basis for a standard.
 - (2) 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 SRMs in ADT, and consideration is being given to a TWA 12 contribution to an ASTM round robin.
- Future plans for TWA 12 suggest that attention should remain in the area of ADT (the surveys indicated that other aspects, such as creep and stress-relaxation, are inappropriate for VAMAS action at the present time), with concentration on completing current work before considering other possible topics.

ACHIEVEMENTS

 A broad survey amongst VAMAS participants evaluated their priorities with respect to the different aspects of the general TWA context (ie creep, stress relaxation, durability, etc), and was responsible for the choice of durability as the current specific topic. A further broad survey examined the availability and use in VAMAS countries of standard tests for ADT, and this led to selection of the two current work topics identified above. A recent achievement is the substantial study of the use of SRMs in ADT by G Zerlaut.

Technical Working Area 13 LOW CYCLE FATIGUE

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OBJECTIVE

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

- A round robin interlaboratory comparison has been completed with participation from sixteen European and ten Japanese laboratories.
- Four materials representing three classes of cyclic strain behaviour were tested at three levels of strain range and at two temperatures. Three methods of measuring strain were used and a total of 61 data sets (each containing 9 data points) have been analysed.
- Evaluation of the data led to the following conclusions:
 - (i) the interlaboratory scatter was large
 - (ii) scatter due to surface finish of the testpiece, fluctuations in temperature and to different criteria of failure were small
 - (iii) the greater degree of variability in the strongest materials is believed to be due to errors arising from measurement of the small plastic strains involved. Further analysis is being carried out to assess the extent to which material variability is a factor.

Technical Working Area 14

THE TECHNICAL BASIS FOR A UNIFIED CLASSIFICATION SYSTEM FOR ADVANCED CERAMICS

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OBJECTIVES

- To identify and assess the issues inherent in the development of a classification system for advanced ceramics, including consideration of the purpose and scope of the system, terminology and nomenclature and relationships between existing systems and a newly developed scheme.
- To establish a building-block classification structure suitable for international use in a compendium of ways, including trade schedule listings, industrial indicators, statistical tabulations, standards categorisations and materials properties databases.
- To develop mechanisms and institutional links for system implementation that interleave national and international standards organisations and the world's advanced ceramics industrial community.

- The membership of the Technical Working Group, now numbering 16, has been specially formulated so that the participation includes at least two from each VAMAS member nation, representing the national interests of industry, academia and standards organisations. From the membership and other sources a worldwide (1200 entries) list of WHO'S WHO in advanced ceramics and standards has been assembled to provide a conduit for working group outputs.
- An international survey was conducted to assess industrial classification needs and practices. About 250 survey questionnaires were circulated to advanced ceramics producers and users in the VAMAS countries. Approximately 125 responses were received; analysis of results is under way with a final report anticipated by January 1991.
- Existing classification systems on conventional materials have been reviewed; this, in combination with the results of the international survey, gave guidance on a skeletal classification scheme and a "working definition" for advanced ceramics. The envisaged classification system will allow incorporation of fore-runner product types to advanced ceramics, generally termed "engineering ceramics" and "technical ceramics". Accordingly, the term "advanced ceramics" should be properly called "advanced technical ceramics".

 A classification workshop was held 21-22 June 1990 at the CECs JRC-Ispra (Italy) to assess classification models developed thus far and to provide recommendations for the best system logic and final structure. A report on the workshop will be published by January 1991.

VAMAS Calendar

VAMAS Steering Committee Meeting: Capri, Italy

1 - 2 November 1990

QSA-6, International Conference on Quantitative Surface Analysis Royal National Hotel, Russell Square,

London, UK (TWA 2 meets on 13 December)

National Symposium of American Vacuum Society 37th - Toronto, Canada 38th - Seattle, USA 13 -16 December 1990

8-12 October 1990 11 -15 November 1991

ECASIA-91, 4th European Conference on Applications of Surface and Interface Analysis Budapest, Hungary

14 -18 October 1991

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