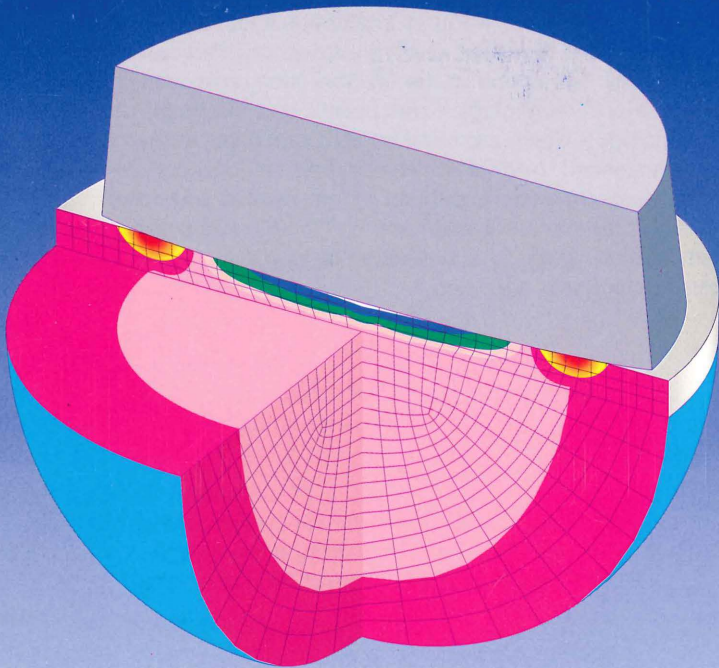




VAMAS



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Versailles Project on Advanced Materials and Standards  
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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 - databases, test methods, design methods, and materials technology - which are required as a precursor to the drafting of standards for advanced materials. VAMAS activity emphasises collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardisation action. Through this activity, VAMAS fosters the development of internationally acceptable standards for advanced materials by the various existing standards agencies.

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Cover: Illustration of a high modulus nano-indenter in contact with an aluminium layer on sapphire showing the vertical displacement field. The contours range from 16.6 nm (white) to 6.9 nm (red). The spherical surface is constrained to conform to the displacement field which would be generated by an equivalent point force on the axis.

*Illustration courtesy of NPL*



# VAMAS

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## ● Foreword ●

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Over the past 10 years VAMAS has gained a mature and recognised status for its role in providing a sound technical and harmonised approach to the development of standards for advanced materials. This has been reflected in the new MoU which operates for an unlimited period from 1997, provided that member countries continue to support the agreement. The new agreement has already been signed by the majority of the VAMAS member countries and the remainder are expected soon to follow.

Within an ever increasing movement towards the global economy, the Steering Committee is keen to stress that VAMAS is not exclusive to the signatory countries and participation of organisations which can make a contribution to a particular project is strongly supported. Indeed, the participation list for VAMAS projects already extends to many countries outside the group of signatory nations and the EU. In practice, applications for participation in VAMAS are formally approved on a case by case basis by a TWA co-ordinator, who is a member of the Steering Committee. Further to this aim, informal contact is also being maintained with ICMET, the International Centre for Materials Evaluation Technology, based at KRISS, the Korean Research Institute for Science and Research, and set up under the auspices of UNIDO (United Nations Industrial Development Organisation).

Since the inception of VAMAS, industrialisation of economies throughout the world has increased at a remarkable rate bringing a greater awareness of the need for sustainable technologies that contribute to minimising the environmental impact of economic growth. In support of this movement, VAMAS and ISO are negotiating with CTI, the Climate Technology Initiative of OECD/IEA, to sign statements of intention to assist in the development of materials standards related to climate friendly technologies. The Steering Committee is currently reviewing potential new activities and will be placing areas in support of the CTI/VAMAS agreement high on the agenda.

I am also pleased to report the first project within VAMAS that has crossed TWA boundaries. This new project is a combined effort between TWA1 and TWA7 and is aimed at using relevant methodologies in each TWA to develop test methods for characterisation of wear debris from artificial human joint materials and for assessing the toxicity effects of differing wear debris morphologies on tissue cultures (see article in this issue).

Finally, to those readers who have noticed the extended gap between this edition of the Bulletin and the last, I should mention that it is now planned to publish on an annual timescale.

Kamal Hossain  
Chairman

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## ● New VAMAS Website ●

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We are pleased to announce that the VAMAS site on the World Wide Web is now up and running. It can be accessed on:

<http://www.npl.co.uk/npl/vamas>

The inaugural pages give a description of the aims and objectives of VAMAS, its structure and brief details of the areas of work. The contents are set out as follows:

1. Formation and Objectives
2. Structure
3. Secretariat and Steering Committee National Representatives
4. Technical Working Areas
5. Publications
6. Links with International Standards Bodies and Other Organisations

Links have been set up with the parent organisations of the National Representatives on the Steering Committee, including direct E-mail access. It is also possible to work down through the general description of Technical Working Areas (TWAs) to explanations of the work of each area, including the contact details for the TWA Chairman.

It is intended to develop the site further, although the level of detail that will be provided on the home site is still under discussion. The Steering Committee is keen to utilise the World Wide Web as a convenient and efficient means of publicity and promotion and is encouraging each TWA to set up its own site, linked to the home pages.

Starting with this issue, the Bulletin will be accessible through the World Wide Web. Potential areas for further expansion will be a list of national representatives for each TWA, details of VAMAS Technical Reports, other publications based on VAMAS work and a list of standards that have been derived wholly or in part through the pre-normative research activities of VAMAS.

The Secretariat would be grateful to receive any helpful comments on the VAMAS Web site which should be directed to the Secretary (contact details in the last section of this Bulletin).

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## ● Standards Highlights ●

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

Standards Highlights identifies draft or adopted standards documents from national, regional, or international standards bodies that are based all or in part on technical outputs from VAMAS TWAs. In the absence of a central standards clearing house, VAMAS participants are strongly encouraged to notify the Secretariat of any such adopted standards and to send a copy to the VAMAS Secretary.

Recent highlights include:

ISO/CD 13003

**Fibre Reinforced Plastics: Determination of Fatigue Properties under Cyclic Conditions**

VAMAS contributor - TWA5, Polymer Composites, Dr G D Sims.

ISO/CD 12106

**Axial Strain Control, Low Cycle Fatigue Method**

VAMAS contributor - TWA13, Low Cycle Fatigue, Dr F A Kandil.

ISO/TTA2

**Tensile Tests for Discontinuously Reinforced Metal Matrix Composites at Ambient Temperatures**

VAMAS contributor - TWA15, Metal Matrix Composites, Dr C Masuda.

IEC TTA No 2

**Critical Current Measurement Method for Nb<sub>3</sub>Sn Multifilamentary Composite Superconductors**

VAMAS contributor - TWA16, Superconducting Materials, Dr H Wada.

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## ● Feature Article ●

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### CONCLUSION OF ACTIVITY TWA 4 Multiphase Polymers

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#### ***From Polymer Blends to Multiphase Polymers: Evolution of objectives***

Activities of the VAMAS Technical Working Area 4 were launched formally at the first meeting of the grouping, at the NRCC in Montreal, in April 1985 under the chairmanship of Dr L A Utracki. At that time there was a great deal of scientific activity and optimism regarding the commercial potential of **engineering** polymer-polymer blends. The complete lack of standardised characterisation procedures, in all structural and performance aspects of these novel materials, was perceived as a significant barrier to applications and international trade. The TWA therefore undertook the task of ***'co-ordinating and carrying out the required pre-normative research in five technical areas: (1) melt flow, (2) dynamic testing of solids, (3) thermal analysis, (4) morphology and (5) mechanical properties in the solid state'***.

In Phase I a co-ordinator for each technical area was appointed, with the responsibility for sample distribution, supervision and reporting on the work in the particular discipline, carried out in all of the VAMAS countries. (The present author was the technical coordinator for the mechanical property measurements over the entire lifetime of the TWA and took over the Chairmanship of the TWA in 1993.)

With considerable support from the polymer blends industry, work was started on a series of model blends of polycarbonate (PC) and linear low density polyethylene (LLDPE), which was to serve as an example of the extreme case of a completely immiscible polymer-polymer blend. This series of materials provided an illustration of the range of problems encountered in the processing, characterisation and testing of this kind of two-phase polymeric system [1-4]. Attention was focused on the particular difficulties in characterising the variable microstructure in the samples [5] and on the determination of the fracture resistance [6-8].

Phase II of the work programme was to ***'examine the limits of applicability of the test methods identified in Phase I'***. The work concentrated on commercial grades of partially compatibilised blends of engineering polymers, namely those of polyamide

(PA) with polypropylene (PP), and on a well compatibilised blend of PA with poly(phenylene ether) (PPE). Again, all the materials were donated by the respective materials suppliers and the industry covered much of the cost of the processing and shipping of the considerable quantity of samples.

There were no apparent problems in the testing of the well compatibilised blend, over and above any of the concerns relating to the testing of polymers in general [9-10]. The expected complications concerning effects of moisture absorption in polyamide systems were noted [11]. In contrast, the partially compatibilised PA/PP blend proved very difficult both in the processing and in characterisation. There was a very pronounced dependence of the morphology in the solid state upon the processing conditions in the melt state [12] which rendered any simple descriptions of structure meaningless and many mechanical tests in the solid state invalid [13,14].

As the extent of the task of developing useful standards for these complex materials became more apparent, so did the need for funded effort. Much of the underpinning industrial research had concentrated on the development of the proprietary compatibilisers. The reasons for this were clear - the use of a suitable compatibiliser ensures reasonable structural stability of the blend during processing and avoids some of the most difficult problems of material anisotropy. However, the early commercial optimism regarding engineering polymer blends was beginning to show itself as unjustified and the inevitable cut-backs in R&D as well as in the product ranges followed.

The rapid decline in the industrial involvement in the early nineties was paralleled by a reduction in the voluntary effort on the part of the largely academic community of test-developers. Efforts were made to combine some activities of the TWA with other standards directed groupings. A successful example of a synergistic combination was a connection with the European Structural Integrity Society (ESIS). The Technical Committee 4 on Polymers and Composites has been working on the development of fracture mechanical standards since 1985 [15]. Its first product is scheduled to become an ISO standard in the near future [16]. As a result of the combined activity between ESIS TC4 and VAMAS TWA4 there will follow an appendix to this standard for the specific application to multiphase polymers [17].

The period of transfer of chairmanship of TWA4 coincided with a re-evaluation of its objectives, in light of the reduced industrial support. There was a broad consensus that the resources of TWA4 should be concentrated upon investigating the relationship between the morphology in the (post-processed) solid state and the high-strain mechanical behaviour in that state. It was felt that there was useful methodology in existence already, namely in the characterisation of anisotropy in mouldings made from short fibre reinforced thermoplastics. There was also a re-evaluation of the methods for 'Design data acquisition for plastics' underway in some of the VAMAS countries. An attempt was made to achieve some synergy through synchronisation of the separate activities. To this end the scope of TWA4 was widened to include the industrially important short fibre reinforced thermoplastics (SFRP) and the name was



changed accordingly, to '**Multiphase Polymers**'. A new set of objectives was agreed in 1994, as follows: (a) *to provide the technical basis for drafting standards test procedures for quantitative morphology analysis, suitable for those polymeric materials which produce heterogeneous structures when moulded* and (b) *to provide inputs into standardisation of test specimens and the mechanical testing methods required to develop a design data base for such polymeric materials* [18].

A round-robin test on the quantitative evaluation of morphological parameters was initiated, using multiple copies of a single photograph of the (relatively simple) structure of the well compatibilised PA/PPE polymer blend used in the earlier work. One of the most frequently used structural parameters, such as the areal proportion of 'particles' on the photograph, were found to vary by as much as a factor of 4, between the eleven groups taking part in the study [19]. The selection of binarisation thresholds in the digital image analysis is of crucial importance. This raises serious doubts on the current ability to compare structural information between laboratories, let alone on the general validity of any predictive models for the mechanical behaviour of multiphase polymers. Concurrent efforts on the development of a fracture test for SFRPs also concluded that no further refinements could be contemplated until there exists an agreed method for morphology characterisation [17].

#### **Advances towards standardisation: 1985-1996**

"Why do we need specific standards for polymer blends?" was the recurrent question in the early years of the TWA. This was based on the, not entirely unreasonable, assumption that a satisfactory and comprehensive bank of test methods was already available for 'polymers'. The fact that this is not the case was obvious to those trying to characterise the raw materials, make large and complex mouldings and design engineering structures from polymer blends. To this date it is not clear which ISO committee within TC61 should receive and deal with any proposals for standards specifically suited to blends, in the technical areas covered by TWA4. Nevertheless, it was the very lack of standards and consequent lack of confidence in the design data which, together with the economic recession, put a stop to the applications and further development of the ambitious, tough, partially compatibilised polymer blends in the early nineties.

The approach taken by the ESIS TC4 in the development of a standard for Kc/Gc fracture testing of polymers (ISO/DIS 13586, ASTM D 5045) [16] points to a viable way forward: an existing methodology standard (from the field of metals testing) was taken and adapted for use with 'simple' polymeric materials in the first instance. There followed a further development of an appendix to the test protocol, in order to specify the exclusions and limits of applicability to multiphase polymeric systems. When this appendix is completed and joined to the main body of the protocol, it will represent the best available method for fracture mechanical testing of materials such as short glass fibre reinforced plastics and those polymer blends exhibiting structural anisotropy on the scale of micrometers [17]. Other fracture mechanical tests are in the process of development, some are well advanced. There is no reason why they should not also be adaptable for use with the more complex polymeric materials, as appropriate [14].

The last decade has seen progress in national and international standards developments in most of the other technical areas highlighted by TWA4. For example, different parts of ISO/6721 cover different aspects of dynamic testing of polymers such as dynamic mechanical thermal analysis, torsional tests and rotation rheometry (ISO/DIS 6721 Pt 10). The developments have been based on relatively simple, structurally homogeneous, polymeric materials. In the case of some test methodologies, there may be no need to make specific exclusions and amendments on the basis of structural inhomogeneity of a material. However, other parameters may assume a greater importance, such as for example the effects of preconditioning upon the apparent values of transition temperatures in some polymeric systems [11]. Recent developments in experimental techniques such as 'modulated' differential scanning calorimetry may make it reasonable to re-visit the appropriate standards with good prospects of solving some hitherto inaccessible problem areas, relating to transition temperatures and recrystallisation in polymers.

If the approach taken by the ESIS group to standards development in polymeric systems is considered a suitable model to follow, then any future method specific standards may need to be linked to *material (type) specific* standards for exclusions and amendments.

The technical area in which standardisation has progressed the least is morphological characterisation. The results of the TWA4 round robin [19] make a compelling case for a concentration of effort in this field. At the present time there are rapid advances taking place in all aspects of computerised data acquisition and analysis. However, there is a lack of definition of the specific requirements to optimise these new techniques for application in complex heterogeneous systems exhibiting limited optical contrast, such as multiphase polymers.

### ***Perceived future needs: recommendation for a new TWA***

Most properties of multiphase polymers depend on the morphology, in the molten and in the solid states. For solid state testing the specimens must be prepared by one or other of the standard polymer processing routes: injection moulding, extrusion or compression moulding. The combination of the nature of the polymeric system and of the processing conditions determines the degree of anisotropy which develops in the sample. The implications of the structural anisotropy are primarily in the accompanying anisotropy in the mechanical properties. However, the mechanical performance reflects the global anisotropy of the sample, without the ability to resolve the effects on the structural scale of micrometers. There is no substitute for microscopy. Quantitative microscopy is perceived to be the essential tool in developing standardised morphology characterisation procedures for multiphase polymeric systems. In turn, the valid and standardised characterisation of the morphology is seen as the essential tool in further developments of mouldings for standard test specimens and of the test methods themselves.

In the light of this perspective the closure meeting of TWA4, held at BAM in Berlin in February 1996, recommended the establishment of a new VAMAS TWA. This new grouping should build upon the background outlined here, but to limit its objectives to the establishment of an international standard on quantitative morphology determination.

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# ● Feature Article ●

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## **NEW TECHNICAL WORKING AREA TWA 22 Mechanical Properties of Thin Films and Coatings**

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### **1 INTRODUCTION**

Thin films and coatings are widely used in a large number of industrial sectors (microelectronics, optical, engineering, aerospace, automotive, biomedical and power generation, for example) thus the industrial impact is especially strong and surface engineering is generally recognised as providing high added value. A major consideration in the design of a coated component is mechanical compatibility between the substrate and the coating, so that it is important to have valid measurement methodologies to generate the required mechanical property data. At present there are a number of empirical and/or semi-quantitative (ranking) standards for adhesion, but otherwise only a handful of other standards for hardness and residual strain [1]. A good example of the quality of current standards is hardness where measurements made during indentation are believed to reflect only the coating properties if, for a hard coating on a soft substrate, the penetration depth is less than one tenth the coating thickness [2] or less than one third for a soft coating on a hard substrate [3]. These empirical “rules of thumb” are without scientific justification and are clearly unsatisfactory. This TWA was established, therefore, to carry out the research required to develop fully quantitative standards.

At present two projects have been approved. The first, on the use of depth sensing indentation (DSI) for the measurement of hardness and Young’s modulus, started in April 1996 and the second on adhesion measurements was initiated about one year later. The forum for discussion of work in this area is the annual International Conference on Metallurgical Coatings and Thin Films (ICMCTF) held in San Diego.

### **2 DEPTH SENSING INDENTATION - HARDNESS AND MODULUS**

Hardness and Young’s modulus measurement were chosen as the first topic on an opportunistic basis because there had been a number of recent international initiatives developing DSI calibration procedures and in conducting intercomparisons with bulk

materials, so that much of the essential preliminary work had been completed [4,5]. DSI is a relatively new measurement method in which an indenter penetrates the sample either under load or displacement control during which the load and displacement are monitored during loading and unloading. The resultant indentation is a measure of the plastic deformation (hardness), while the elastic response (Young's modulus) is derived from the slope of the unloading curve (Figure 1). Instruments are now available which cover the load range of conventional microhardness indenters (minimum load ~100mN) to the nN range, while minimum displacements of a few nm are possible for the highest resolution instruments.

## **2.1 Objective**

The purpose of the project is to carry out research to allow the development of appropriate standards for the measurement of hardness and modulus of coatings. This will be achieved by an investigation into the measurement and analytical methodology used during indentation tests and in the analysis of results to derive hardness and modulus values of the coating. Thus the work aims to answer the question of the one tenth or one third rule referred to above, and essentially there are two ways of proceeding. The first is somehow to determine the depth at which the substrate begins to contribute to the measured hardness (there will always be a contribution from the substrate for Young's modulus measurement) or to 'deconvolute' the coating properties from the measured composite values. In the latter case this is usually accomplished by numerical modelling, but recently some analytical models have been developed which claim both elastic and plastic solutions [6-8]. Indeed, with the increased usage of coatings only a few tens of nanometers in thickness, use of an appropriate model is the only possibility since it will not be practicable to indent to a depth where only the coating hardness is measured.

## **2.2 Work Plan**

To initiate the work two coating systems have been agreed: aluminium deposited by electron beam evaporation onto sapphire and, the 'inverse', an amorphous alumina deposited by reactive magnetron sputtering onto an aluminium substrate. Sets of samples covering the thickness range 0.1 - 5  $\mu\text{m}$  have been produced and distributed to over 60 individual organisations. A work plan to investigate seven parameters has been agreed and experimental work is now progressing with over twenty sets of results already produced. The experimental variables and the range being investigated are listed in Table I where it can be seen that the effects of loading rate, load range, hold time at maximum load, indenter geometry, repeated indentations and coating type and thickness are being investigated. An important consideration in planning this work has been to limit the amount of experimentation by each participant, and in general only about a minimum of 1 - 2 days effort has been required, although some participants have carried out additional work.

## 2.3 Calibration

Before starting on the experimental work, it is important that all instruments are calibrated for load, displacement, instrument stiffness and indenter area function. The need to calibrate for load and displacement is clear, but perhaps a word of explanation is needed for the instrument stiffness and indenter area function calibrations. All indentation experiments involve monitoring displacement during loading of the specimen, and if during this operation displacements also arise from deformation within the structure of the instrument serious errors can arise, so that a suitable correction to the measured data must be made. The instrument compliance correction is usually derived by carrying out indentations into bulk reference materials (usually fused silica and aluminium are chosen) over a range of loads. The compliance for the system - sample and instrument - is then calculated from the unloading slope (Figure 1) and then the instrument compliance determined from the intercept at zero load of a plot of total compliance versus load.

The indenter area function calibration is required because many instruments have the capability of measuring displacements of only a few nanometers, and at that scale one cannot make the assumption that the indenter has the theoretical shape. If, as is usual, a Vickers or Berkovich indenter is used then some rounding at the tip is always found, similarly for conical or spherical indenters which are also sometimes used, spherical symmetry is seldom observed. Thus it is necessary to determine the projected area of the indenter as a function of distance from the tip of the indenter, and both indirect methods (indentation into reference materials) [9] and direct methods (for example, AFM [10] or replica methods [9]) are used. Participants in the first phase of the work have been asked to detail their calibration procedures and also to carry out the same experimental tasks in bulk fused silica as well as in the coated sample. In this way it will be possible to separate calibration errors from the effects of the instrument variables associated with the coated samples.

## 2.4 Results

### *Analysis*

Analysis of loading and unloading curves to obtain hardness and Young's modulus using depth sensing indentation is also not entirely straightforward. In fact the DSI community has adopted a new definition of hardness, **H**:

$$H = \frac{P_{\max}}{A_c}$$

where  $P_{\max}$  is the maximum load and  $A_c$  is the projected contact area at **maximum** load. (The normal definition of hardness is the maximum load divided by the projected area of the **residual** impression). Young's modulus is defined from the slope of the unloading curve as:

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A_c}$$

where  $h$  is the displacement and the reduced modulus,  $E_r$  is defined as follows

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}$$

and  $E$  and  $\nu$  are, respectively, the Young's modulus and Poisson's ratio of the specimen and  $E_i$  and  $\nu_i$  corresponding properties of the indenter. The major challenge is associated with determination of  $A_c$  where there are still largely unresolved difficulties. The current method is to fit the unloading curve to the following form:

$$P = A (h - h_f)^m$$

where  $A$ ,  $h_f$  and  $m$  are obtained by a least squares fitting approach. The initial (elastic) unloading slope is then found by differentiating this expression and determining the tangent at maximum load. Extrapolation of this tangent to zero load is then the contact depth for a flat punch indenter, but further modification is required for other indenter shapes. Once  $h_f$  has been determined,  $A_c$  can then be derived from the indenter area function. As will be inferred from the previous discussion the procedure is not straightforward and considerable care is required in both the calibration and analysis of the data if accurate results are to be obtained.

#### *Experimental data*

Figures 2 and 3 show, respectively, typical loading/unloading curves for the alumina and aluminium coatings. It is noteworthy that for the alumina coating (hard on soft) the loading curve shows a distinct change in curvature and if, instead of plotting a direct function of load and displacement, load is plotted versus the square of displacement this change in behaviour is shown more clearly (Figure 4). Hainsworth et al [11] have suggested that this critical load corresponds to plastic deformation of the substrate. Likewise Rother et al [12] have proposed that the differential of the load displacement curve plotted against displacement will also indicate the onset of substrate effects, and Figure 5 shows this plot for the data shown in Figure 2. While both these 'empirical' methods suggest a procedure for determination of the load at which the substrate begins to influence the measured hardness or modulus, there is, as yet, no sound theoretical basis for the method.

### **3 ADHESION**

The second project that is being planned is on the determination of adhesion. As mentioned in the introduction, there are many different test procedures that have been proposed for the measurement of adhesion. The approach being adopted here therefore is to have one test which may have quite wide applicability that will be studied by all participants while each is also free to introduce their own 'favourite' test method. Four-point bending has been chosen as a method which should allow determination of interfacial shear strength of brittle coatings on ductile substrates, and this is to be the test used by all participants. As with the work on DSI a major effort is required on modelling four-point bending.



Coatings typical of the engineering and electronics sectors are to be studied. Coatings are now being produced and work is expected to start later this year.

#### 4 CONCLUSIONS

Work of this new TWA is now established and the prospects for future projects seem secure since there are a number of other important mechanical properties of thin films and coatings for which either no internationally agreed standards exist or standards are inadequate. Important areas for future work include residual strain, fracture strain, toughness, ductile or brittle transition temperatures and thermal mechanical fatigue. In these areas, as with adhesion, there are various possible test procedures, each of which may be better suited to different types of coating/substrate system. Thus there is a large body of work which is required to put the measurement of mechanical properties of thin films and coatings on a sound metrological basis.

#### 5 REFERENCES

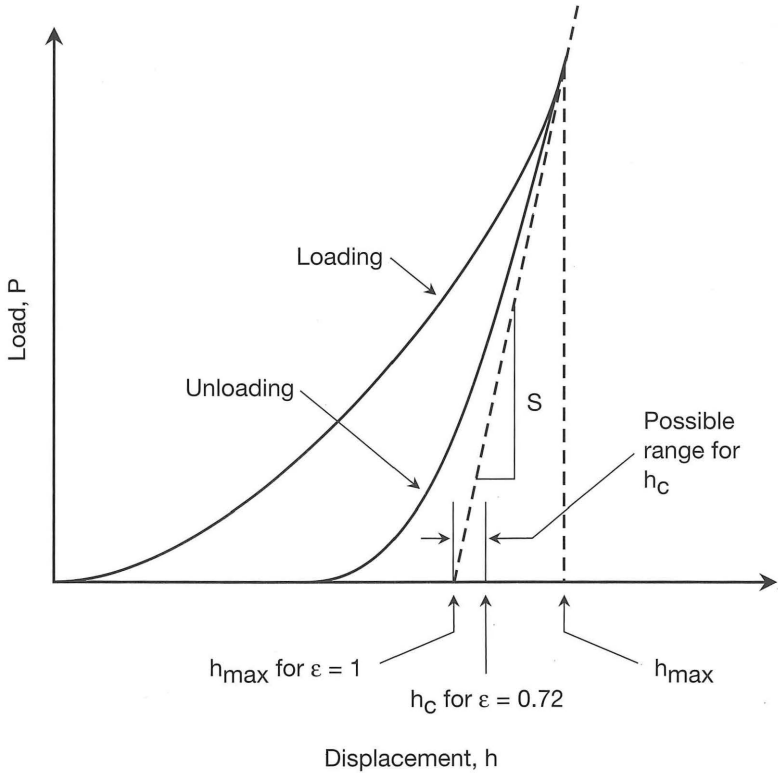
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- 2 BSI Standard: BS 5411 Pt6 *Metallic and Related Coatings, Part 6 Vickers and Knoop Microhardness Tests* Committee ST/37, and ASTM Standard: B578-87 -reapproved 1993 Microhardness Testing of Electroplated Coatings
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Table 1: Experimental Work

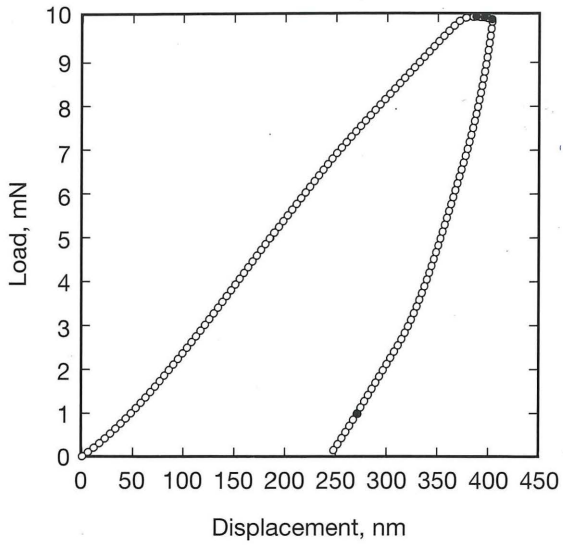
Coating type	Coating thickness, $\mu\text{m}$	Task	max Load / load range, mN	Loading rate, $\text{mN s}^{-1}$	No of cycles per indent	Hold time, s	Estimated max displacement, * nm	No. of Indents
Alumina	all	Coating thickness- Task 1 (5 samples)	10.0	0.1	1	30	400	50
	1.0	Loading rate - Task 2	10.0	0.01, 0.1, 1.0 ,(10)	1	30	400	40
	1.0	Coating type - Task 3	1.0	0.1	1	30	80	10
	1.0	Loading range - Task 4	0.1, 1.0, 10, 30, 100	0.1	1	30	0! - 3000!	50
	1.0	Indenter geometry ** - Task 5	10.0	0.1	1	30	400	30
	1.0	Repeat loading - Task 6	10.0	0.1	1, 2, 3, 4, 5 (10 indents per experiment )	30	400	50
	1.0	Hold time at max load - Task 7	10.0	0.1	1	1, 3, 10, 30, 60	400	50
Aluminium	all	Coating thickness - Task 1 (3 samples)	1.0	0.1	1	30	300	30
	3.0	Loading rate - Task 2	1.0	0.01, 0.1, (1.0 ,10)	1	30	300	40
	3.0	Coating type - Task 3	1.0	0.1	1	30	300	10
	3.0	Loading range - Task 4	0.01, 0.1, 1.0, 10	0.1	1	30	0! - 1000	40
	3.0	Indenter geometry** - Task 5	10.0	0.1	1	30	1000	30
	3.0	Repeat Loading - Task 6	1.0	0.1	1, 2, 3, 4, 5 (10 indents per experiment )	30	300	50
	3.0	Hold time at max load - Task 7	1.0	0.1	1	1, 3, 10, 30, 60	300	50

\* based on bulk materials using a Berkovich indenter

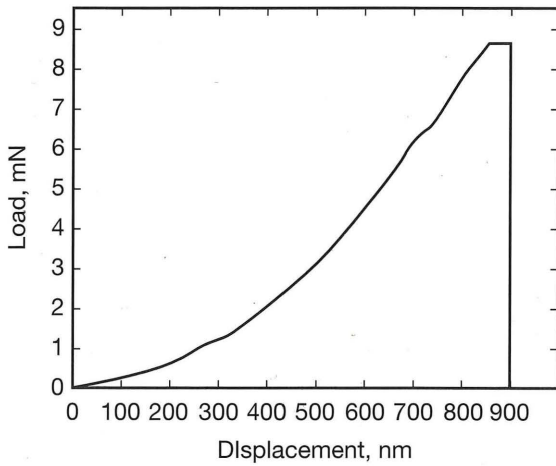
\*\* Specify indenter shapes and area functions



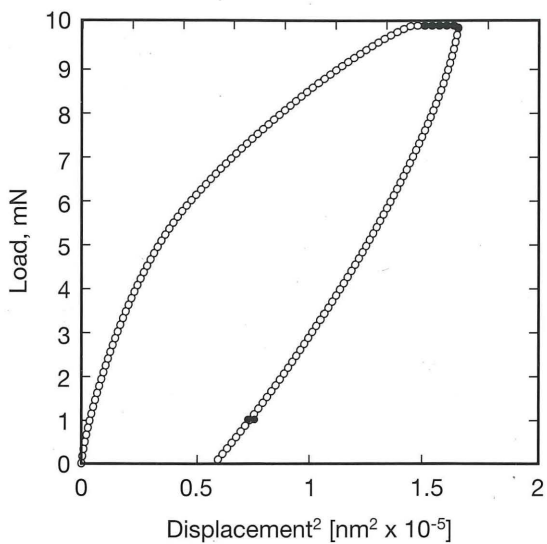
**Figure 1** Schematic representation of a load displacement curve (after ref 9)



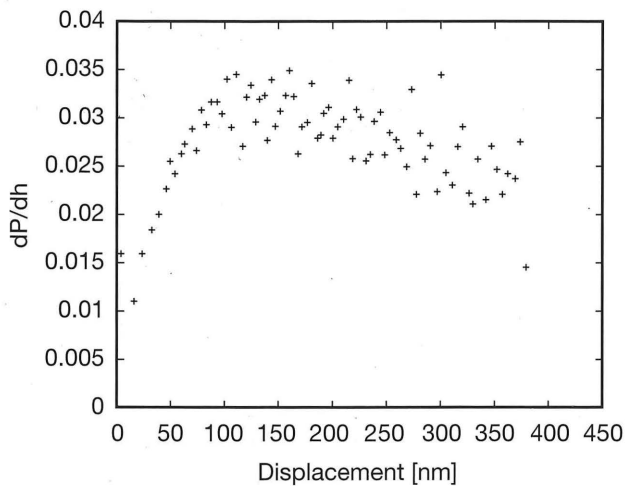
**Figure 2** Load/displacement curve for 1µm alumina coating deposited on aluminium



**Figure 3** Load/displacement curve for 3µm aluminium coating deposited on sapphire



**Figure 4** Load/displacement squared curve for 1µm alumina coating deposited on aluminium



**Figure 5** Differential load/displacement curve for 1µm alumina coating deposited on aluminium

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# ● Recent VAMAS Outputs ●

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## EXTENDED ABSTRACT

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VAMAS Technical Report No 22  
"Bend Strength Measurements for Hardmetals -  
International Prestandardisation Collaborative Activity.  
Part 1 - Rationale and Results"  
by B Roebuck,  
Centre for Materials Measurement and Technology,  
National Physical Laboratory, UK

This report provides a compendium of results of an international collaborative activity on bend strength measurements for hardmetals, designed to re-evaluate the current standard for Transverse Rupture Tests (bend tests) for Hardmetals, ISO 3327. Developments in the understanding of strength measurements, the increasing desire for data more relevant to material quality and design and the need for a test method to give results comparable to test data for competing materials, such as ceramics and cermets, led to agreement to perform this interlaboratory exercise and the formation of TWA21 (see leading article in VAMAS Bulletin No 20).

The purpose of the exercise was to examine a number of alternative methods for bend tests on hardmetals to that specified in ISO 3327, to include: geometries that compare with standards for ceramics, tests on small specimens, unconventional geometries, and possibly also methods of specimen preparation.

The interlaboratory tests involved fourteen laboratories, mostly industrial, from eight countries testing seven materials using eleven testpiece geometries. Preliminary examination of the data indicated that very good agreement was obtained between tests at different laboratories, although considerable variations in measured strengths were recorded for the different geometries. Testpiece preparation method was also noted as an important factor and notched specimens gave less scatter, unless the material was processed to contain few macroscopic internal defects.

A further report is planned to analyse the data in more detail and to make specific recommendations for amendments to the ISO standard.

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# ● Recent VAMAS Outputs ●

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## EXTENDED ABSTRACT

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VAMAS Technical Report No 23

"A Unified Classification Scheme for Advanced  
Technical Ceramics"

Testing and Demonstrating the VAMAS Classification Scheme,  
Summary of Issues in Manual Coding of Commercial Products  
by R Morrell, Centre for Materials Measurement and Technology  
National Physical Laboratory, UK

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I Stamenkovich, Centro Ceramico Bologna, Italy

R Wäsche, Bundesanstalt für Materialforschung und -prüfung,  
Berlin, Germany

Following the success in carrying the VAMAS unified classification scheme for advanced technical ceramics into the standards arena (ISO TTA No 1, GEN ENV 12112, ASTM C1286-94), the next step was to provide a basis for acceptance by the industry for which it had been primarily developed. Phase II of the work of TWA14 was therefore devoted to testing the scheme on commercially available materials and components and to developing a computer programme which could demonstrate a number of potential applications. This report describes the testing of the scheme.

Over 300 commercially available products were examined and coded by four organisations with information obtained from data sheets supplied by various European countries and the USA. Coded data from a separate Japanese test of the scheme were also included in the survey. Any problems associated with coding the information were documented and collated in the report and are summarised as follows:

- The coding problems resolve into two types:
  - the specific code is not available, or not obvious
  - insufficient/incorrect explanation for unambiguous choice of code.
- A considerable number of minor revisions need to be considered.
- The major issues identified are:
  - clarification of the scope and purpose of scheme to avoid attempts to classify in excessive or unnecessary detail.
  - 'applications' for fine ceramic powders is unclear.
  - the use of short-form codes for powders is unclear.

- there may be a need to include coding for some aspects of component performance data, as distinct from material property data.
  - the language describing the meaning of a code may be unclear or too general.
- Minor issues are:
    - missing codes.
    - inconsistencies in code descriptions.

This report contains a detailed listing of the above issues, and gives some possible solutions to the problems. These issues need to be agreed by standards committees (CEN, ASTM and ISO) as proposed modifications to standard documents.



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# ● Recent VAMAS Outputs ●

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## EXTENDED ABSTRACT

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VAMAS Technical Report No 24

"Classification and Designation Systems for Materials"

A report on the present situation, an inventory of the systems in use, and comments on future possibilities

by K W Reynard, Wilkinson Consultancy,  
Newdigate, Dorking, Surrey, UK

Technical Working Area 10 (TWA10), Materials Databanks, recognised in VAMAS Technical Report No 2 "Factual Materials Databanks: The Need for Standards", the benefits to be gained by rationalising existing materials classification and designation systems. They recommended that as a first step the existing systems should be catalogued and this report is a response to that recommendation.

The report gives a summary of the diverse systems that are in use for the major classes of engineering materials and includes an inventory of national and international standards that give specifications for materials used in many applications and also some standards and codes of practice that give recommendations for presentation of materials data.

Comment is included on the increased knowledge on and the greater demand for data that affect materials performance, the increasing use of computerised materials data and the risk of accepting those data with limited knowledge of their origin. Recommendations for future activities in the standardisation of classification and designation systems are given in Section 9 of the report.

[Note: Section 10 which contains 245 pages on the Inventory of Standards is contained on a floppy disk provided as an attachment to the report.]

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# ● Recent VAMAS Outputs ●

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## EXTENDED ABSTRACT

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VAMAS Technical Report No 26  
“Morphology Quantification of Multiphase Polymers”  
by T Nishi and T Takayuki, University of Tokyo, Tokyo, Japan

To quantify the effect of alloying and alloy process conditions on polymer properties it is essential to understand the relationship between structure and properties. Digital image analysis (DIA) is potentially a powerful method for structural analysis but it is a relatively new technique which has been developed in different countries using various types of equipment and applying non-standardised methods.

This report describes the results of a round robin conducted by TWA4 using the same transmission electron microscope (TEM) images of a poly(phenylene ether) (PPE) and polyamide (PA) blend that exhibited a two-phase structure of spherical island particles in a sea. Eleven organisations from five countries (Belgium, Canada, France, Germany, Japan, United Kingdom) took part to establish initially the degree of variation in the results from the different methods and to identify the key parameters that need to be considered in the development of a standardised procedure.

The participants were requested to make measurements in two categories: particle analysis and spatial distribution of particles on a similar alloy processed under 2 different extrusion conditions and at 2 magnifications, ie 4 separate micrographs. No conditions on the measurement technique were imposed on the participants.

A wide variation was found to exist in both DIA systems and in the methodologies employed. Results obtained by different groups were also very different from each other. Whilst DIA itself clearly has high potential for analysing morphology in a quantitative manner, there appears to be a complete lack of standardisation in the procedures involved. Some of the important summaries from these round robin studies are as follows:

1. It is essential to show the binarised images and the binarisation method.
2. For the analysis of spatial distribution of particles, there seems to be no standard and it is very important to show the binarised data and explain the definition of parameters or functions to describe the distribution.

TWA4 has now ceased its activities but discussions are taking place to form another TWA to investigate specifically morphology characterisation (see feature article).

## **SURFACE CHEMICAL ANALYSIS**

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### **Objectives**

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

Activity in TWA2 continues apace with the development of new projects, projects undertaking interlaboratory studies and with material feeding through to ISO/TC 201 on Surface Chemical Analysis.

One new proposal for work has been made since last year. This is entitled 'Reference Coatings for evaluation of GDOES' (Glow Discharge Optical Emission Spectroscopy) and was sent out for comment on 1 April 1997 for discussion at the June meeting in Goteborg. This will be our first effort in GDOES which is an important industrial analytical technique. It is likely that the films used in this work could help other projects too. It is anticipated that work will start on this project in January 1998.

Work on the project on characterisation of oxygen vacancies which has been active at the national stage has been inhibited by commercial considerations. This should change in the near future as SFV (French Vacuum Society) seeks to promote co-ordinated work by proposing a Charged Dielectrics Division for IUVSTA (International Union for Vacuum Science Technique and Applications).

Of the three new projects reported in the last Bulletin the first two are gathering momentum and the one dealing with evaluation of static charge stabilisation and determination methods in XPS on non-conducting samples has requested participants to respond using the form of ISO 5725 (Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests). This ensures that those who offer to participate have considered the relevant resources required and provides a sound basis for the ensuing work. Samples have been sent to all participants with a response required by the end of the year. The third of these three projects, the interlaboratory study of static SIMS repeatability and reproducibility is complete and a report has been presented. This has shown that repeatability can be as poor as 50% where in-house procedures are ill-defined. However, where control is good, the samples are sufficiently consistent for 1% to be demonstrated.

## CERAMICS

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### Objective

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

1. The National Industrial Research Institute and the Japan Fine Ceramic Center have coordinated a project on conventional hardness of ceramic composites. This project features Vickers 9.8 N and 98 N, and Knoop 9.8 N and 19.6 N indentations in several orientations in hot-pressed SiC whisker-reinforced  $S_3N_4$  composite specimens. An enormous amount of data (over 2000 hardness readings from 21 laboratories) have been analysed, leading to some preliminary conclusions:
  - a) Hardness decreased with increasing indentation load, which is normal for ceramics. The scatter decreased at higher loads.
  - b) Variation in hardness with whisker orientation was masked by the (usual) scatter in results. The effect of indentation orientation and indented surface was small.
  - c) Hardness increases with an increase in whisker content. Scatter was similar for monolithic and composite specimens.
  - d) Good consistency between laboratories (reproducibility) and the lowest scatter within laboratories (repeatability) were obtained with the Knoop 2 kg procedure but only 14 of 21 participating laboratories had the capability to create indentations at this load.
  - e) Conventional hardness measurement methods are suitable for ceramic composites.

The draft of the final report is currently being reviewed by participants.

2. A second ceramic hardness project has been coordinated by the Federal Institute for Materials Research and Testing (BAM) in Berlin, in cooperation with the National Institute of Standards and Technology, USA. In the low-load testing regime, there is considerable interest in measuring depth of penetration simultaneously with the application of load. BAM organized a project to study the Recording (Instrumented) Hardness of Ceramics. A BK-7 optical glass and a silicon nitride hardness reference block are being used. The latter are prototype Knoop Standard Reference Material blocks prepared by NIST. Participants were asked to monitor displacement of Vickers and/or Berkovich indenters at a prescribed loading rate with maximum forces of either 1 N or 10 N. This round robin finished in 1997 with only two laboratories in the United States not able to return their results and a draft final report is being reviewed by the participants.
3. The quantitative microscopy project is a joint undertaking of the Center for Technical Ceramics, the University of Eindhoven and the National Physical Laboratory, United Kingdom. The program is a joint VAMAS and CEN Technical Committee TC184, Advanced Technical Ceramics program. Participants applied both manual point analysis and automated image analysis (AIA) methods to characterize the second phase content and porosity of a computer-drawn microstructure, photomicrographs of several real microstructures, and an actual ceramic specimen. All results have been furnished to the organisers who have prepared the initial tabulations and preliminary conclusions. A final report is in preparation.
4. The draft final report on a completed project on fracture toughness of ceramic composites is completed.
5. A new TWA3 project, the fifth to feature fracture toughness, commenced in July 1997, Fracture Toughness by the Single Edged V-Notched Beam Method (SEVNB). The SEVNB method is a refinement of the earlier single-edge notched beam and single-edge V notched beam methods, except that a very sharp notch is introduced by a razor blade and diamond paste. The method was described initially by Nishida, Hanaki, and Pezzotti, *J. Am. Ceram. Soc.*, 77 [2] (1994) pp 606-608. This project is a joint European Structural Integrity Society (ESIS) - VAMAS TWA3 project. Thirty-six laboratories are participating. This project is popular since the test method purports to be a simple, practical way to obtain a precrack. Most single-edge notched beam results are compromised by the notch bluntness. The very sharp razor slit may approximate the sharp cracks needed to obtain valid fracture toughness data.
6. A further project, Surface Roughness of Advanced Technical Ceramics, is under review at the moment as a possible VAMAS exercise. A round robin on this topic has already commenced under the auspices of CEN TC184. If accepted this project will be coordinated by the National Physical Laboratory, United Kingdom and will feature diamond stylus profilometer characterization of as-fired, ground, lapped, and polished ceramic components.

## **BIOMATERIALS/WEAR TEST METHODS**

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### **Objective**

- To develop test methods suitable for evaluating biocompatibility of hard tissue materials by means of evaluating toxicity of wear debris in vitro by using cultured cells.

### **BACKGROUND**

Total joint replacements in human bodies are increasing in quantity world-wide. This is due to increasing life expectancy, population growth, advances in medical science, rigorous exercise, and an increasing number of accidents. One of the key life-limiting factors of the current bio-components is the generation of numerous particles from the wear of ultra-high molecular weight polyethylene (UHMWPE) in contact with the cobalt-chromium (CoCr) alloy.

A project within TWA1, under the leadership of Dr Stephen Hsu at NIST, is undertaking an international round robin study on wear debris characterisation methodology and representation. The material system to be studied is an ultra-high molecular weight polyethylene and a cobalt-chromium alloy. The objective of the study is to develop wear debris characterisation methods in terms of size, shape, and morphology, and to develop mathematical representations of the debris. While the wear debris formation is the current life limiting factor of artificial joints, the exact nature of the linkage between debris size, shape and morphology to bioactivity is not well understood.

TWA7 under the leadership of MEL in Japan has developed a bioculture technique to determine the relationship between debris particles and bioactivity. The joint TWA1 and TWA7 activity will identify if a particular population of size, shape or morphology is more bioactive than others. This may lead to international standardisation of biomaterial test methods in the future.

## **PLAN**

CoCr alloy and UHMWPE samples will be distributed to participants. Unidirectional polishing methods will be specified for the CoCr alloy surface. Different surface textures will be specified to generate a particular size, shape and morphology of the UHMWPE wear debris. A pin-on-disk wear tester will be used for reciprocating motion in distilled water (or bovine serum, a choice made by individual participants). The ASTM F-732 method will be used as a guide for testing. The focus will be on the collection of wear debris and the analysis of the size, shape and morphology of the debris thus generated. Part of the debris generated will be sent to the TWA7 participants for biocompatibility testing.

## **INTERNATIONAL PARTICIPATION**

Twenty-one laboratories in eleven countries have agreed to participate in the TWA1 wear activity. We are still seeking laboratories to conduct bioculture tests. Interested parties should contact Dr Tateishi to join the TWA7 activity on the bioculture test round robin.



## **HIGH TEMPERATURE FRACTURE OF BRITTLE MATERIALS**

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### **Objectives**

- Developing a test method for determining the creep crack growth rate of creep brittle materials.
- Understanding material behaviour both in terms of mechanics and micromechanisms.
- Dissemination of information by open workshops and symposia.
- Feeding the conclusions into standards-making bodies so that valid test standards can be generated.

TWA19 has had an active year. Two full meetings were held; one in March in Sydney, Australia, which was associated with ICF7 (International Conference on Fracture) and the other in October in Kyoto, Japan. Further presentations were also made to report the findings of the project at the Spring and Fall meetings of ASTM (American Society of Testing and Materials). The next meeting is expected to take place in London in March 1998.

From a previous project in TWA11, procedures were developed for measuring the creep crack growth properties of materials for creep ductile situations using the creep fracture mechanics parameter  $C^*$ . Details of the method are contained in ASTM standard E1457-92. Restrictions are imposed in this standard to ensure that data are only collected after steady state creep stress and damage distributions have been developed ahead of a crack tip for valid correlation with  $C^*$ . The present investigation was initiated to examine to what extent the procedures specified in E1457 can be applied in the presence of limited creep deformation.

In TWA19, a series of round robin creep crack growth tests has been performed on three materials having different creep characteristics. Data have been collected on a

C-Mn steel at 360 °C, a titanium alloy at 500 °C and an aluminium alloy at 135 °C under conditions where only limited creep deformation was measured. Although analysis of the results has still to be completed, it has been established that modification to E1457 is required before it can be applied to situations involving limited creep deformation. After discussions with ASTM in committee, it has been agreed that the ratio of creep to total displacement rates that are specified to accompany cracking can be relaxed from the present 0.8 to 0.5 for valid data still to be produced. This change will be included in the next issue of E1457 to allow it to cope with more brittle circumstances. It is likely that further suggestions for revision will be made at the conclusion of TWA19.

It is planned that the main findings of TWA19 will be published in a special issue of Engineering Fracture Mechanics in 1998.

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## ● VAMAS Calendar ●

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- IVC 14, 14th IUVSTA International Vacuum Congress  
incorporating QSA10, International Conference on  
Quantitative Surface Analysis, Birmingham, UK ..... 31 Aug-4 Sept 1998
- PSA 98, International Symposium on Practical Surface  
Analysis, Matsue, Japan ..... 19-21 Oct 1998
- 45th AVS, Baltimore, USA ..... 2-6 Nov 1998
- SIA 98, Asia-Pacific Surface and Interface Analysis  
Conference, Singapore ..... 30 Nov-4 Dec 1998
- TWA3 (Ceramics) Meeting in conjunction with CIMTEC  
Ceramics Conference, Florence, Italy ..... June 1998
- TWA5 (Polymer Composites) Meeting in conjunction with  
CTS/4, Lisbon (provisional) ..... 31 Aug-2 Sept 1998
- TWA13 (Low Cycle Fatigue) Meeting at NPL,  
Teddington, UK ..... Sept 1998
- TWA16 (Superconducting Materials) Meeting in  
conjunction with the ASC, Palm Desert California ..... Sept 1998
- TWA19 (High Temperature Fracture of Brittle Materials)  
Meeting in London ..... Mar 1998
- TWA20 (Measurement of Residual Stress) Meeting at  
NIST, USA ..... Apr 1998
- TWA22 (Mechanical Measurements of Thin Films & Coatings)  
meeting in conjunction with the International Conference  
on Metallurgical Coatings and Thin Films, San Diego, USA ..... 27 Apr-1 May 1998

Anyone wishing to join a meeting should contact the relevant TWA Chairman.

# ● VAMAS Technical Working Areas ●

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## **Technical Working Area 5 Polymer Composites**

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## **Technical Working Area 13 Low Cycle Fatigue**

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## **Technical Working Area 14 Unified Classification System for Advanced Ceramics**

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## **Technical Working Area 16 Superconducting Materials**

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## **Technical Working Area 17 Cryogenic Structural Materials**

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## **Technical Working Area 18 Statistical Techniques for Interlaboratory Studies and Related Projects**

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# ● VAMAS Technical Working Areas ●

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## **Technical Working Area 19 High Temperature Fracture of Brittle Materials**

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