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# Advances in Materials Technology: MONITOR

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MATERIALS DESIGN AND EVALUATION

Dear Reader,

This is number 33 of UNIDO's state-of-the-art series in the field of materials entitled *Advances in Materials Technology: Monitor*. This *Monitor* is devoted to **MATERIALS TESTING AND EVALUATION**.

The main article for this *Monitor* was written for us by M. K. Hossain, National Physical Laboratory, Teddington, Middlesex, United Kingdom.

We invite our readers to share with us their experience related to any aspect of production and utilization of materials and especially comments on the subject of the *Monitor*. It would be appreciated if you could answer the questions on the "Reader Survey", to be found at the end of this *Monitor*, and return it to us. Thank you for your cooperation.

Technology Development and Promotion Division

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# 1. TESTING AND EVALUATION OF ADVANCED MATERIALS

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## INTRODUCTION

Industrial competitiveness, economic growth of nations, and the quality of modern life all rely strongly upon innovations in materials technology and hence it is considered widely to be a critical technology. Without new and improved materials and processes, recent advances in fields such as computers, communications, transport, health care and leisure could not have been realized. By the turn of the century markets for advanced materials have been estimated to reach \$400 billion globally, and in the USA alone 1.5 million "high skill" jobs will depend upon new developments in this field<sup>(1)</sup>.

Materials evaluation and testing plays a central role in successful development, but more importantly, in the applications of new materials into products. In this paper key issues related to testing and analysis are discussed, with particular reference to structural materials i.e. materials with load bearing properties. The importance of standardization of testing methods is considered in the light of globalization of markets and of industrial manufacture. Much effort and resources are needed to meet the challenge and there are significant opportunities for co-operation amongst countries in underpinning work to provide the technical base from which widely acceptable standards can be developed. An analysis of the current situation with indications for future developments is presented here.

## VALUE OF TESTING AND EVALUATION TO INDUSTRY

Many of the important advantages of advanced materials may be attributed to their superior performance. Market requirements tend to dictate their use in more severe operating conditions, in areas of greater added value and in products emphasizing greater design efficiency than traditional materials. All of these require reliable information on materials behaviour and properties based on sound materials evaluation techniques. Thus, reliable testing and evaluation methodologies are required for:

- (a) Material development,
- (b) Quality control and assurance,
- (c) Materials specification,
- (d) Engineering design,
- (e) Product development and assessment,

- (f) Determination of residual life, and
- (g) System maintenance.

Requirements will vary in individual cases, but it follows from the above that information or data on a range of material parameters may be called for, e.g. in the case of structural applications of engineering materials this could cover:

#### Mechanical Properties

Stiffness, strength, creep, toughness, impact, fatigue, wear etc.

#### Environmental Properties

Resistance to heat, light, humidity, corrosive medium (gaseous or liquid)

#### Surface Properties

Composition, structure, adhesion etc.

#### Process-related Properties

Viscosity, surface tension, cure, heat transfer etc.

In addition, measurements of electrical, optical, magnetic or a combination of these properties may be required for certain applications and materials.

In the industrial context, materials evaluation is relevant to all the key stages of manufacture. Based on an identified market need, the designer prepares a design brief using which preliminary ideas about a product (schematics) are developed. At this stage materials selection, selection of manufacturing methods and first order cost estimate for the product are made. These are based on relatively straightforward test data or materials properties and often a family of materials is chosen.

The next phase is concerned with detailed design and developing manufacturing specifications. To undertake this work, the structural engineer must have reliable and high quality materials property data and appropriate design analysis methods. Standards and codes of practice are sometimes available providing guidance on specific types of component design, the codes of practice may specify standardized test methods which must be used to obtain properties data. For detailed design, particularly for critical applications, suitable materials data can often only be obtained by testing materials made by the chosen manufacturing process and in the anticipated environment. Structural testing of the component/product may also be required to fully validate the design and production method. Finally, in the manufacturing phase, tests to comply with quality control and assurance are carried out including non-destructive evaluation (NDE).

Evident from the above is the fact that materials property data are required at various stages, but the nature and quality varies according to whether one is dealing with materials selection, design or production.

In Europe, USA and Japan, the importance of testing and evaluation has been clearly recognized. Thus, the Presidential Materials Initiative launched in the US in 1992 contains materials characterization including assessment of performance, properties and structural relationships as one of the four key components<sup>(2)</sup>. In Japan a large industrial survey has concluded that priority in standardization should be given to testing and evaluation methods<sup>(3)</sup>. Similarly, a 1991 study of the European Commission on Industrial and Materials Technologies: Research and Development Trends and Needs, confirmed that performance testing and standardization should be a priority theme for medium term R&D<sup>(4)</sup>.

### ISSUES IN MATERIALS EVALUATION

For high pedigree design data, advanced materials often require different approaches to materials testing based on a sound understanding of their structure and behaviour. In many cases validated measurement methods do not exist with the consequence that there are disagreements about interpretation of results and unwillingness to accept others' results. Accuracy, meaning how close the measured value of a property is to the real value, is a particular problem because real values cannot be calculated or established otherwise. Areas for particular concern are:

- Anisotropic nature of many materials and products;
- Microstructure and behaviour can be complex relative to conventional materials;
- Final properties depend upon processing route and conditions;
- Demanding test conditions e.g. high temperature, complex environments;
- Absence of suitable design methodologies which dictate actual measurement requirements;
- Increasing need for extrapolation of data;
- Requirement for wider range of data and data of much higher accuracy than previously;
- Physical measurement techniques are being used near their current limits.

**Testing of Composites** Some of the points may be best illustrated by considering, say, testing of composite materials. Components made from composites are often complex and tailored to provide strength and stiffness specifically where these are needed. Such anisotropy and inhomogeneity give rise to immediate complications for materials testing. Even for a relatively simple task as tensile strength measurement, serious problems can arise from non-alignment of test pieces, means of stress transfer and the effects of residual stress and stress concentrations.

For instance, smooth and hard surfaced composites cannot be clamped in the same way as metals because conventional gripping can fracture the matrix and cause failure. End-tabs bonded to the testpiece are used, but these can themselves cause failure in some cases. Another method for overcoming the problem is to drill holes in the testpiece and use pegs to transfer the stress, but this can lead to a non-uniform stress distribution.

The shape and size of the testpiece also requires careful consideration to ensure that the results are representative of the bulk materials as opposed to the testpiece. In tensile testing of traditional materials, testpieces are waisted so that failure occurs in the thinner more highly stressed part of the sample. Adoption of this procedure, say, for a unidirectionally laid up composite with fibres running along the length of the testpiece, means the testpiece has to be I-shaped. Much of the clamp force on the short fibres terminating at the waist is transferred to the matrix so the testpiece fails by cracking at the shoulders of the I.

#### **Creep Testing of Polymeric Materials and Modelling**

Measurement of creep in polymeric materials cannot be treated in the same way as metallic materials due to the physical ageing effects in polymers. After processing, continuous increases in density are observed for most solid polymers due to molecular rearrangements associated with the approach to structural equilibrium. This physical ageing process leads to pronounced decreases in properties such as creep rate, impact strength, dielectric constant and loss factor. Hence, for polymers, it is vital to take into account elapsed time after processing (t) for measurement of creep properties.

Figure 1 shows the variation of compliance,  $D(t)$ , at  $23^\circ\text{C}$  with  $\log(t)$  for polypropylene samples of different age;  $D(t)$  is defined as the time dependent longitudinal strain divided by the constant uniaxial applied stress. At long times, the experimental compliances, (shown as points), are considerably lower than the values obtained by extrapolations (broken lines) of short-term data which have neglected further ageing during the test period. However, if polymer ageing is taken into account using a model developed at NPL<sup>(5)</sup>, the predicted compliances shown as continuous curves, are within 3% of the observed value. This agreement is valid for 2-3 decades beyond the short-term region.

Thus, if a polymeric component is subjected to a constant load one month after processing, its deformation may be confidently predicted for times greater than 10 years from tests of about 1 week's duration. A model based on known physical principles associated with accurate traceable measurements can indeed provide a cost effective answer to the measurement problem. In fact, to characterize the creep of a polymer, a minimum of some twenty five sets of measurement is required<sup>(6)</sup>. For data covering a useful temperature range increases this to several hundred measurements. When one considers that a single basic polymer can exist in the market in many hundreds of different grades the magnitude of the testing task is formidable and models such as above can come to real rescue!

#### **High Temperature Testing**

There is considerable potential for applications of advanced ceramics in structural applications such as gas turbines where higher operating temperatures are needed to improve fuel efficiency. The materials must be able to withstand stresses at temperatures as high as  $1500-1600^\circ\text{C}$  in hostile environments for extended periods of time whilst maintaining a reasonable tolerance of existing or newly created defects. The high stiffness and damage intolerant behaviour of ceramics, coupled with the requirement for testing at very high temperatures, poses specific problems such as:

- Measurement of uniaxial properties without introducing bending stresses;
- Measurement of small strains at high temperatures;
- Establishment of testpiece temperature; and
- Development of furnaces with a sufficiently uniform temperature distribution.

#### **Low Cycle Fatigue**

LCF can be a life-limiting factor in critical components such as pressure vessels and turbines, so testing for LCF is carried out extensively in industry to characterize materials performance and to provide design data for components operating under fluctuating loads and temperatures. Although very similar procedures are now being used world-wide, the variability in LCF data from different laboratories is rather large. This can be best illustrated by the results of an intercomparison exercise by twenty-six well known laboratories in Europe and Japan<sup>(7)</sup>. Materials used were Nimonic 101 at  $850^\circ\text{C}$ , and AISI 316L Stainless Steel, 9 Cr - 1 Mo Steel and IN 718 at  $550^\circ\text{C}$ , all materials being sourced and characterized centrally. Guidelines were provided to define the test variables and each laboratory used its own procedure for conducting the tests.

Figure 2 is a typical example of lifetime dataset showing fatigue life for Nimonic 101 as a function of

th: nominal total strain range. Data from individual laboratories are plotted in separate segments to indicate repeatability (variation within a single laboratory using the same operator) whereas reproducibility (variations between laboratories with different operators) is represented by the overall spread in the "box plot". Good repeatability but poor reproducibility are evident, the worst scatter in reproducibility being a factor of about 50 - which is most unsatisfactory for the designer. Analyses of these results identified the following sources of primary error:

- (a) Misalignment or bending of the testpieces;
- (b) Errors in temperature measurement and/or control;
- (c) Errors in strain measurement and control.

Before the testing problem can be solved satisfactorily, it is necessary to establish the sensitivity of each of the above parameters on the final result. Further work has shown that the effects of variation in strain measurement are quite small. As regards bending, a model has been developed which provides a quantitative relationship between testpiece bending and fatigue life measurement. Prediction made by the model compares well with experimental data<sup>(8)</sup> as shown in figure 3 confirming that bending is indeed a major contributory factor. Further work is required to develop a best practice guideline for LCF testing that will be based on harmonized procedures for the measurement of load misalignment and testpiece bending, design of testpieces and fixture, and assessment of the effects of temperature variation.

**Engineering Coatings** Coatings are applied in many industrial systems where surfaces of bulk materials suffer problems, particularly due to wear or corrosion. Industry often uses simple specifications e.g. a coating of certain minimum thickness and known porosity, but there is a lack of reliable evaluation methods and adequate standards even for the basic specification. Widely differing methods are sometimes used where results are not easily comparable and cannot be interpreted unambiguously. A recent study of industrial requirements in Europe has identified the need for new or improved test methods for measuring:

adhesion,	corrosion resistance,	porosity,
thickness,	friction/wear resistance,	internal stress,
surface finish,	microhardness,	and fracture toughness.

Figure 4 compares, for each property, its perceived intensity of need against our current level of knowledge. Thus, coatings adhesion to the substrate is considered to be one of the most important parameters to establish whilst accurate determination of adhesion is

extremely difficult. For weaker bonds (80-100 MPa), pull tests, lap shear, double cantilever beam, napkin ring and moment methods with adhesives give useful results, but care must be taken to align and establish the failure mode (i.e. adhesive and not cohesive).

For stronger bonds, it is common practice to use the scratch test, but the analysis of results is by no means straightforward and there is considerable debate about the value and validity of the test method<sup>(9)</sup>. New techniques, including a four point bend test and nanoindentation, are being developed for measurement of adhesion.

### FUTURE TRENDS IN TESTING AND MEASUREMENT

Changes in materials testing and assessment are being dictated strongly by advance in design methodologies and quality initiatives. Traceable data of greater precision and reliability are required by industry, but since good quality data are scarce and expensive, innovative materials models are emerging that enable greater use of the data. Similarly methods for reliable extrapolation of short-term test data to predict long-term service performance and techniques for correlation of simpler test results to performances in complex conditions have increased in demand.

Mechanical testing equipment is becoming more complex - computers are used not only to operate the machines but also to collect, evaluate and process the output data with the minimum of operator intervention. This, however, means the operator must have good knowledge of the test technique and data processing, particularly the limitations, to ensure the pedigree of results. Software validation in this context has become a truly important issue.

More severe test environments and conditions are being required, e.g. very high test temperatures approaching 1800° C and above are increasingly needed for testing intermetallics, ceramics and composites. Environments which chemically interact with the material are also being superimposed to predict behaviour in service conditions. In addition, there is a growing drive for more bi-axial and tri-axial testing to simulate complex loading in components.

As discussed earlier, specimen bending can give rise to large errors in mechanical testing such as fatigue life assessment and modulus measurement. Therefore, test machine manufacturers are placing greater emphasis on the design of frames with increased lateral stiffness. Self-aligning grips and sensors for the measurement of bending of testpieces are also available for advanced evaluations.

The demand for servo-hydraulic testing machines in which materials can be tested under high rates of



application of load or strain is increasing. Future trends for these machines are towards advanced control strategies with adaptive control using feedback from measurement and sensing devices. Improvements are being sought in the calibration of load cells and extensometers under dynamic conditions.

Currently measurement uncertainties are not regularly quoted, which can pose difficulties for the users of test results. The situation is changing with pressure from the standards and accreditation bodies. For instance, it is the declared policy of ISO and the European standardization authority that all new standards concerned with testing techniques shall contain "a statement of uncertainty" or a method of calculating the accuracy of the test method based upon tolerances specified in the relevant standard.

Determination of remanent life of large industrial systems such as power generation and petrochemical plants is now frequently sought in order to decide whether their operational lifetime can be extended beyond the design lifetime, which obviously has large financial implications. Generally, fracture or component damage and distortion due to excessive deformation, wear or corrosion leads to loss of function and therefore, for predictive purposes a host of materials properties data such as creep, low cycle fatigue and wear in the presence or absence of corrosive environments are needed. Important to note here is the fact that the data precision level required is much greater than historically demanded for design purposes and so more stringent testing is required.

Improved techniques for quality control and non-destructive testing and evaluation (NDT) are increasing in demand. Thus, industry requires improved methods for the evaluation of mechanical properties (e.g. bonding of composites), higher-resolution defect detection, improved computer tomography, sensing devices for physical and chemical properties of materials.

**Measurements related to materials processing** There is a growing recognition that improvements in the processing of advanced materials is of utmost importance. Test techniques that can be used for process design and control are, therefore, in urgent demand. Major customers for engineering components are beginning to insist on process qualification rather than product inspection as a philosophy for quality assurance. Sophisticated mathematical models are being increasingly used as an important tool by industry to improve processes and to "achieve right first time" (see figure 5) - application of such modelling has advanced enormously due to the availability of computing power and software packages that benefit even quite small producers.

However, these models require reliable data for the physical properties associated with the heat and fluid flow of the processes which are not readily available. This is because appropriate measurement techniques are

not available. For example, a recent analysis has shown that for plastics, important problem areas for measurements include:

- (a) Viscosity,
- (b) Elasticity, and
- (c) Fibre orientation and distribution.

Viscosity is perhaps the most important property with two aspects to be measured - shear viscosity and extensional viscosity - each is a function of temperature and flow rate.

The most common method of measurement of shear viscosity for polymer melts is a capillary rheometer with dies of various lengths and diameters. End corrections are made in analysing the data and generally satisfactory results can be obtained. Difficulties arise in measurement of shear viscosity in relation to high rates and high viscosity. Measurements for extensional viscosity, on the other hand, is not as well developed. When it comes to measuring elasticity there is a great deal of confusion and much more work is needed before reliable measurements could be made and data incorporated in models.

In the case of metallic materials and processes, new or improved methods are required for measuring:

<b>heat flow</b>	
heat capacities enthalpy melting range	thermal conductivity heat transfer properties
<b>fluid flow</b>	
density viscosity	surface tension thermal diffusivity

Many of the processes operate at high temperatures where interactions between the material and its test container can cause serious problems. It is not surprising that data in the published literature are prone to large uncertainties and frequently there is no information available for commercial materials. At NPL a new programme of work has been initiated to provide the necessary data and methods required for industrial applications of models used in casting, rolling, forging and moulding.

#### REQUIREMENTS FOR STANDARDIZED TEST METHODS

Testing requirements for an advanced material is dictated by its application. However, because there is a lack of standardized methods, users often develop their own test methods to characterize and qualify the material

and establish design allowables. Thus, despite the fact that the need for basic materials properties may be similar for a group of users, they use different methods for pragmatic reasons and expediency. As a result, a major supplier of composite materials is known to undertake up to ten variations of essentially the same test to prove the integrity of its materials to its various customers, many of which are large companies in the Aerospace sector each imposing its own individual specification where a common specification would suffice. Multiple test methods lead to ambiguities and inefficiencies with consequent penalties for materials users, suppliers and test houses alike.

From the market point of view, the principle disadvantages are:

- Unnecessary duplication of work by materials suppliers and users;
- Customised test facilities are expensive to build and operate;
- Test results cannot be compared easily;
- Databases for common use are difficult to develop.

Indeed, the lack of widely accepted test methods and data impedes the market use of these materials in new areas. What industry needs is common or international product standards incorporating harmonized materials evaluation/specification methods since this will facilitate trade and reduce the need for multiple testing.

#### DEVELOPMENTS IN INTERNATIONAL STANDARDIZATION

Materials technology is advancing rapidly making it difficult to set standards for measurements and testing. Although continual progress is being made in relating materials characteristics to properties and performance, the quantitative basis from which widely acceptable standardized methods can be developed are not available in many areas.

International standardization is considered to be a relatively slow process and, traditionally, has been active after the market for new products has been established to allow fair competition. Necessarily this has meant international standardization activities have concentrated on harmonization of existing national standards.

In 1990 the international standards bodies ISO and IEC initiated a study by a team of top industrialists and businessmen to review standardization needs for rapidly developing technologies such as materials, IT and biotechnology. Their report - A Vision for the Future - contains a stimulating analysis of the potential

contribution standards can make in the technological innovation process<sup>(10)</sup>.

Different aspects of standardization relate to the different stages of innovation and product development as follows:

State of technological development	Type of standard necessary	Main benefits
Early establishment phase	Nomenclature and terminology	Improved communication in the market, collection of statistics.
Growth phase	Measurement and test methods	Design, production and reliability of products; enhance markets.
Well established	Product standardization	Rationalization, interface capabilities, market protection etc.

For standards which enhance "market entry" of products based on new technologies, it was recognized that international standards bodies needed to take new steps in addition to their traditional consensus activities. These should be:

- Flexible (i.e. direction setting agreements or provisional quasi-standards)
- Fast (i.e. very simple procedures)
- Open (i.e. to direct participation from companies and/or national organizations with R&D programmes).

Recommendations of the above report are being implemented with strong support from the ISO/IEC councils. In fact, as a result, three new technical committees in the advanced materials field have been established recently. A list of important ISO committees currently active in the materials field is given in annex I.

#### STANDARDIZATION IN EUROPE, JAPAN & USA

**Europe** Dramatic changes are taking place in Europe where measures to develop a Single Market and to open up public procurement amongst the European Community members depend critically on the availability of European standards. These standards are

mainly developed by CEN (European Committee for Standardisation), ECISS (European Community Iron and Steel Standards) and CENELEC (European Committee for Electrotechnical Standardisation). CEN and CENELEC have concluded agreements with ISO and IEC respectively for regular discussion of their work programmes with a view to avoiding overlap and, furthermore, international draft standards are considered for adoption by CEN wherever possible (see figure 6).

Membership of European Standards Committees is open to EC and EFTA (European Free Trade Agreement) countries, currently 18 altogether. An important feature of European standardization is that while agreement on technical contents is reached by consensus, adoption of the standard is by weighted majority voting. European standards must be adopted as national standards, regardless of the way the national member voted, and any conflicting national standards have to be withdrawn.

In materials CEN's activities are divided into four areas:

- (i) Iron and steels products,
- (ii) Non-ferrous metals,
- (iii) Non-metallic materials, and
- (iv) Other materials.

A list of the standards committees<sup>(11)</sup>, their scope of activity and standards already developed is given in annex II. ECISS with a long history has the most extensive coverage. In the non-ferrous metallic materials area six committees are active dealing with aluminium, copper, zinc, tin, lead and nickel. Between 1988 and 1991 CEN created all the seven committees working on non-metallic materials covering *inter alia* textiles, refractories, plastics and advanced ceramics. In the final area, four committees deal with non-destructive testing, foundry technology, corrosion and powder metallurgy.

**Japan** Amongst developed countries, Japan has one of the most rigorous standardization programmes on advanced materials with strong industrial involvement and participation. An extensive survey<sup>(3)</sup> of standardization needs and feasibility of standards development for industrial use covering the fields of metals, polymers and ceramics was undertaken in 1988 by the Japanese Industrial Standards Committee (JISC). They concluded that nearly seven hundred standards should be developed as a matter of urgency within the next 10-12 years. Of particular relevance here is the recommendation of the Committee that terminology, testing and evaluation methods should be given priority.

Following the recommendation of the committee, a five-year plan was launched in 1991 to promote industrial standardization. Highlight of the plan is

Japan's new policy for greater co-operation with ISO/IEC activities. In fact, Japan is taking new initiatives to advance international standardization at the expense of national and regional standardization.

**USA** Most widely known is the work of ASTM, the American Society for Testing of Materials. Individuals from any country can participate in ASTM committees which rely primarily on voluntary effort by professionals in industry, academia and government. ASTM currently have 120 technical committees covering the following main areas:

A Ferrous metals	B Non-ferrous metals
C Cementitious ceramic, concrete and masonry materials	D Miscellaneous materials
E Miscellaneous subjects	F Materials for specific applications
G Corrosion, deterioration and degradation of materials	

#### SOME IMPORTANT STANDARDIZATION ACTIVITIES

**Advanced Ceramics** Applications of advanced ceramics in high technology products require the availability of test and measurement methods for powder properties as well as the properties critical to the design and manufacture of ceramic components. These materials have very different properties and applications compared with traditional ceramics, so the scope for adoption of existing test and evaluation methods developed for, say, porcelain and refractories is rather limited. Since the majority of potential users of advanced ceramics have little experience of how to apply them in products effectively, it is widely acknowledged that reliable evaluation techniques would pave the way for increased application and market penetration.

As early as 1981 saw the first publication of a standard for these new materials by the Japanese who continue to provide a major thrust in this area. The first standard was on "Testing method for flexural strength (modulus of rupture) of high performance ceramics". Since then the Japanese have published ten more standards in English concerned with the measurement of various mechanical, thermal and corrosion properties. The Japanese Industrial Standards Committee has identified<sup>(3)</sup> some 47 items on test methods for priority action (annex III).

In the USA, standardization work in this area commenced in 1985. ASTM and its committee C-28 on advanced ceramics has a comprehensive work programme in the following key areas:

- Properties and performance
- Design and evaluation
- Processing and characterization
- Ceramic matrix composites, and
- Nomenclature.

In Europe, 4/5 years ago, the European Commission made a mandated request to both CEN and CENELEC for the establishment of a comprehensive programme for setting up voluntary Eurostandards (ENV) and European Standards (EN) in the field of advanced industrial ceramics. Accordingly, in 1989 CEN established a Committee, CEN/TC 184, with specific tasks to develop up to forty standards on classification, terminology, sampling and methods of test. The methods of test include physical, chemical, mechanical, thermal and textural properties for ceramic powders, monolithic ceramics, ceramic composites and ceramic coatings. The standards developed by CEN TC 184 and its work programme are shown in annex IV<sup>(12)</sup>.

Internationally, research related to standardization is being carried out under VAMAS (described later) and also under the International Energy Agency Programme by the USA, Sweden and Germany. Based on the VAMAS programme standards have already been developed for wear test methods of ceramics, and further standardization can be expected for testing dynamic fatigue, hardness, fracture toughness at room and high temperature, fatigue and grain-size measurements.

A very important development has come recently from ISO when a Japanese proposal to set up a committee on technical ceramics was endorsed by five ISO member countries. The new committee, TC 206, is being launched with participation from Australia, Indonesia, Jamaica, Japan (Secretariat), Republic of Korea, Malaysia, Russian Federation and USA. A wide ranging programme has been proposed, but the final scope has not been agreed, partly because of lack of participation from Europe and the need to develop a programme that will build upon existing work in Europe and USA. The first meeting of the TC is expected to be held in 1994.

**Polymers and Polymer-Composites** Internationally the drive for harmonization of test methods for polymers and polymer-composites has grown steadily over the past 6/7 years and a number of new initiatives have been

launched. A very important development relates to standards for the presentation of comparable data for plastics in ISO. Manufacturers of polymeric materials supply property data but, historically, different manufacturers have used different test procedures to obtain their data. As a result, users of plastics are faced with the problem that the data available for nominally similar materials from different suppliers are not readily comparable, making materials selection rather difficult. Furthermore, properties of plastic materials depend not only on the molecular structure, and orientation and concentration of any fibres or reinforcements, but they also vary with the test conditions chosen for the measurement, such as time, temperature, loading rate and the stress level. Hence, an adequate characterization of materials and behaviour can be expensive.

In recognition of these problems a series of international standards is being developed that specify test methods, specimen geometries and test conditions to enable comparable data on plastics to be measured. These standards deal with single and multipoint data; the first group relates to a limited range of common properties, each property being described by a single measurement value. These single point data are used for the initial stage of materials selection and are shown below.

Properties included in the single-point data presentation standard ISO 10350	
<p><u>Mechanical</u></p> <p>Tensile modulus Yield stress Stress at break Yield strain Elongation at break Tensile creep modulus Flexural modulus Flexural strength Charpy impact strength Tensile impact strength</p> <p><u>Electrical</u></p> <p>Permittivity and dissipation factor Electric strength Volume and surface resistivity Comparative tracking index</p>	<p><u>Thermal</u></p> <p>Melting temperature Glass transition temperature Temperature of deflection under load Vicat softening temperature Linear thermal expansion Flammability Ignitability</p> <p><u>Rheological</u></p> <p>Melt mass-flow rate Melt volume-flow rate Moulding shrinkage</p> <p><u>Other</u></p> <p>Density Water absorption</p>

Properties included in the multipoint data presentation standards ISO 11403-1, 2 and 3				
Part	Property	Status	Standard	Variables
1	<b>Mechanical properties</b>	Draft international standard		
	Dynamic modulus		6721-2,4	T
	Ultimate values of stress and strain		527-2	T
	Tensile stress/strain curves		527-2	e,T
	Tensile creep strain		899-1	$\alpha$ ,t,T
Charpy impact strength	179	T		
2	<b>Thermal and processing properties</b>	Draft international standard		
	Enthalpy			T
	Linear expansion			T
Melt viscosity	11443	$\gamma$ ,T		
3	<b>Environmental influences</b>	Committee draft		
	Liquid chemicals		175	L,t,T,I
	Prolonged action of heat		2578	t,T,I
	Artificial weathering		4892-2	H,t,I
	Environmentally assisted stress cracking		6252	L,t,T

T - temperature  
H - radiant exposure  
 $\gamma$  - shear strain rate  
e - strain  
 $\sigma$  - stress  
t - time  
I - indicative property  
L - liquid chemical

The multipoint data standard ISO 11403, as described in the preceding table, has three parts. It deals with more extensive data sets on property values as a function of variables such as time, temperature and the environment, and the data, therefore, can be used for later stages of materials selection. In fact, some of these data will be suitable for use in design calculations for predicting performance in service and for selecting optimum process conditions.

The ISO plastics committee (TC 61) has a broad range of activities on unreinforced and reinforced materials, although many of the current test methods in composites are related glass-fibre reinforced materials. Standardization of materials reinforced with carbon and aramid fibres has been slow, but this is changing. Recently, methods for the determination of density and size, and definitions and vocabulary for carbon fibres have been standardized. Furthermore, as existing standards are updated, advanced composites are being included as is the case with ISO 527: Plastics: Determination of Tensile Properties. Also, for example, the revised ISO 1268 - Test Panel Manufacture covers a variety of fabrication routes such as autoclaving, filament winding, pultrusion, spray-up moulding, contact moulding and compression moulding.

Further topics include linear density and twist of fibres, strength of glass fibre yarns and mats, tensile

properties of carbon fibre impregnated yarns and single filaments, curing behaviour of pre-regs or compounds, and tensile strength of laminates. ISO/TC 61 has ten sub-committees with nearly 65 Working Groups:

ISO TC-61 (Plastics) Sub-committees	
Sub-committee number	Area of activity
SC1	Terminology
SC2	Mechanical properties
SC4	Burning behaviour
SC5	Physical-chemical properties
SC6	Ageing, chemical and environmental resistance
SC9	Thermoplastic materials
SC10	Cellular materials
SC11	Products
SC12	Thermosetting materials
SC13	Composites and reinforcements

Standardization for polymer composites has gathered momentum in CEN during the last 3 years. Since the pressure to produce standards is high, consideration is given to adopting ISO standards wherever appropriate. Otherwise adoption or re-drafting of European Aerospace standards or national standards is

favoured as the next step. The CEN Committee on Plastics (TC 249) has five sub-committees as follows:

- SC1 Plastics materials and test methods
- SC2 Composite reinforcements and pre-pegs
- SC3 Semi-finished plastic products
- SC4 Cellular materials
- SC5 Plastics for packaging

Each sub-committee is served by Working Groups; SC2, for example, have five working groups: WG1 is responsible for all small diameter fibres (carbon, glass and aramid), WG2-4 are responsible, respectively, for aligned reinforced thermosets, random reinforced thermosets and all reinforced thermoplastics, except short fibre systems (covered by SC 1). Test methods applicable only to specific materials are covered by WG2-4, but WG5 is responsible for test methods which are applicable generally.

That standardization is needed badly in this area is demonstrated by annex V which contains a list of differing standards currently used by various trade and industrial organizations.

**Standards on Corrosion** An adequate knowledge of the degradation behaviour of a material due to its interaction with the operating medium is vital for the efficient utilization of materials. Such degradation can take different forms; the most well known in this category is corrosion of metallic materials, the cost of which can be very high, 2-4% of GDP, with large impacts on industrial competitiveness, environment and

health and safety. Greater awareness and understanding of the processes and effects of corrosion have led to significant improvements in design and building of corrosion-resistant products and systems as well as protective techniques. It is evident that reliable test and evaluation methods to measure corrosion resistance and the effectiveness of corrosion protection techniques play a very important part in this development.

Not surprisingly there is now quite an intensive activity in developing international standards in this field. In ISO, the most relevant activity is being undertaken by TC 107: Metallic and other inorganic coatings, and TC 156: Corrosion of metals and alloys.

Eight sub-committees operate under TC 107, three of which are concerned with terminology, inspection methods and corrosion tests, and the others deal with various types of coatings: electrodeposited coatings, hot dip coatings, thermal spraying, vitreous and porcelain enamels and chemical conversion coatings. Altogether nearly 70 standards have been prepared so far, the majority covering defined preferred properties of applied coatings. Methods for determination of their properties, thickness and corrosion resistance are included. Sub-committee 7 has the responsibility for developing standards mostly on test methods and procedures.

TC 156 "Corrosion of Metals and Alloys" is primarily concerned with test methods and has 19 participating countries and 27 observer countries<sup>(13)</sup>. There are nine working groups working in the following areas:

Activities of ISO TC 156 (Corrosion of Metals and Alloys)		
Working Group No.	Subject area	Comments
WG1	Terminology	ISO 8044 - defines terms relating to corrosion of metals and alloys
WG2	Stress-corrosion cracking	<ul style="list-style-type: none"> <li>• ISO 6509: determination of dezincification resistance of brass;</li> <li>• ISO 7539: 8-part standard on stress corrosion testing (a standard for Stress corrosion Cracking of Al alloys in preparation)</li> <li>• A standard on corrosion fatigue to be published</li> </ul>
WG3	Atmospheric corrosion	ISO 7441 - bimetallic corrosion in outdoor corrosion tests ISO 8565 - general requirements for field tests
WG4	Classification of corrosivity of atmospheres	Four ISO standards nearly completed (ISO 9223-6) including one on Methods of determination of corrosion rates of standard specimens for the evaluation of corrosivity
WG5	Intergranular corrosion	<ul style="list-style-type: none"> <li>• ISO 9400 - Determination of intergranular corrosion of nickel alloys</li> <li>• A standard on intergranular corrosion of aluminium alloys to follow</li> </ul>
WG6	General Principles for conducting corrosion tests and criteria for evaluation and treatment of results	• ISO 8407 - Methods for the removal of corrosion products from corrosion test specimens
WG7	Accelerated corrosion tests	ISO 9227 - Salt spray tests
WG8	Co-ordination	
WG9	Corrosion testing of materials for nuclear power generation	<ul style="list-style-type: none"> <li>• A standard on aqueous corrosion of zirconium alloys under discussion</li> <li>• Work in progress on Electrochemical Potentiokinetic Reactivation (EPR) test for measuring susceptibility to intergranular attack in stainless steels</li> </ul>

New work programmes on electrochemical test methods and cathodic protection of underground structures and marine corrosion test methods are under development.

In the USA, ASTM committee G-1 is principally responsible for standards related to corrosion testing and evaluation. It has twelve sub-committees which have produced over sixty standards covering a wide range of tests and corrosion conditions<sup>(14)</sup>.

Over the last 2-3 years European activities on standardization in the corrosion area have intensified considerably through the establishment of a committee, CEN TC262 entitled Corrosion Protection: metallic materials. However, collaboration between CEN and ISO is close and indeed in certain areas the work proceeds jointly with ISO.

#### INTERNATIONAL CO-OPERATION IN PRE-STANDARDS RESEARCH

The world's most industrialized countries recognized nearly a decade ago the importance of co-operation in advanced technologies for the creation of employment and economic growth. Accordingly, a new initiative known as the Versailles Project on Advanced Materials and Standards, VAMAS, was launched in 1987 with the underlying aim of removing barriers to trade by encouraging the development of standards from a commonly agreed technical base. VAMAS operates on a cost-sharing principle without any central funds whereby project work is carried out by participants with support from their own countries under existing programmes and schemes. In addition to the G7 countries, the CEC is a member of VAMAS, but many organizations from non-member countries have also participated in specific activities by special agreement.

Over sixty technical projects have been initiated so far covering all the key materials classes; activities include the development and validation of test methods, guidelines, codes of practice, terminology schemes and data handling. Primarily VAMAS is active in pre-standards research necessary for the drafting of standards for advanced materials. Close liaison is maintained with national and international standards organizations to focus the pre-standards activities and also to assist in the transfer of results to standards. As a follow-up to the recommendations made in the ISO/IEC report "A Vision for the Future", ISO has recently signed a Memorandum of Understanding with VAMAS to publish suitable VAMAS outputs as "Technology Trend Assessment" documents to accelerate standards development and to disseminate rapidly methods and guidelines which industry can use in the absence of a standard.

Currently, VAMAS has seventeen Technical Working Areas (TWAs) under five main themes as

shown below. A full list of the TWAs are given in annex VI.

#### VAMAS THEMES

Theme 1	Metals and Metal-matrix composites
Theme 2	Polymers and Polymer-matrix composites
Theme 3	Ceramics and Ceramics-matrix composites
Theme 4	Test Techniques
Theme 5	Materials classification and Data

**Surface Chemical Analysis** Technical Working Area 2 in VAMAS is dealing with surface chemical analysis techniques: Auger Electron Spectroscopy (AES), X-ray Photoelectron Spectroscopy (XPS) and Secondary Ion Mass Spectroscopy (SIMS), all extensively used in many industrial sectors for the development of high technology products and materials. VAMAS became active because of a need to produce widely acceptable reference procedures, reference data and reference materials as a basis for standards for surface chemical analysis. In fact VAMAS took the lead in bridging on-going activities in the USA, Japan and Europe all aimed to overcome similar problems.

In this area of VAMAS, nearly 100 laboratories have been participating from about ten countries. Accuracy of measurements of chemical compositions and concentrations are two key issues in AES and XPS. Figure 7 shows that for XPS, the intensity/energy response function of even one instrument cannot be defined uniquely and are different for two operating conditions<sup>(15)</sup>. Complications arise because spectra from commercial instruments are distorted from the true spectrum by the instrument transmission function which depends on the electron energy and the instrument settings. To calibrate these functions NPL has established a reference spectrometer with traceability to primary Measurement Standards so that true spectra and hence true chemical compositions and concentrations can be found<sup>(16)</sup>.

As a result of this pioneering work, one can now obtain consistent and mutually recognizable analysis of the same specimen in different instruments throughout the world. Furthermore, it is now possible to produce reference databanks for international use. To this end the VAMAS group has already developed a standard data transfer format to aid the process of transferring data from one laboratory to another<sup>(17)</sup>.

Success of the VAMAS programme has led to wider international interest and, in fact, recently ISO have set up a new Technical Committee, TC 201 on

Surface Chemical Analysis, with Japan providing the Secretariat, and the USA the Chairman. That there is wide interest in this area is demonstrated by the fact that ten national standards bodies have become participating members (P-members) and fifteen others have become observer members (O-members). VAMAS has liaison category A with ISO/TC 201 that will ensure full communication between the two activities and the results of VAMAS work will have rapid access to ISO. The sub-committees of TC 201 and their proposed areas of work are given in the annexes VII and VIII.

It is of interest to note here that ISO at the same time has set up a new Committee on Microbeam analysis (TC 202), in which China has taken on the important role of the Secretariat.

**Testing of Advanced Ceramics** In this area VAMAS has been active on a number of fronts. Under TWA1, co-operative work involving nearly 40 laboratories examined wear testing of alumina and silicon nitride which showed the importance of controlling machine dynamics and humidity of the testing environment; the latter was found to have a particularly large influence as a humid atmosphere encourages the formation of lubricating films of hydrated debris thereby reducing wear rates. DIN and ASTM have developed two new standards (DIN 50234 and ASTM G-99) based on the VAMAS work; in addition an Austrian draft standard (ÖNORM M 8125) and a UK guideline have been produced recently. Currently, the TWA is working on the development of (i) a uniform format for presentation of wear data, (ii) quantitative methods of assessment of wear volumes to obtain more accurate wear rates, (iii) a directory of standard wear test methods and (iv) harmonized wear testing method for inorganic coatings.

Other important areas in ceramics are covered in TWA3 and TWA14. TWA3 has been concerned with mechanical properties and microstructural characterization, and examined (i) dynamic fatigue testing, (ii) hardness measurement, (iii) fracture toughness testing, and (iv) measurement of average grain size in alumina. For the last activity VAMAS initiated a round-robin exercise at the request of the CEN Committee TC184 and within a year produced robust information on the use of line and circle methods for grain size and porosity measurements on the basis of work by twenty five laboratories in USA, Europe and Japan. The results have directly led to a European standard<sup>(18)</sup>.

Hardness is often considered to be a useful materials property by industry. Although there are a number of well established techniques for hardness measurement of conventional metallurgical materials, it is not clear whether these techniques are suitable for advanced ceramics. Indentation sizes can be very small and the material around the indentation can crack and spall leading to serious measurement difficulties. Indeed a VAMAS exercise<sup>(19)</sup> on two high-alumina ceramics using Brinell HR 45N, Vickers HV 1.0 and 0.2, and

Knoop HK 0.2 tests showed that when indentation sizes are as small as 15  $\mu\text{m}$  and the position of the corners on the indentation cannot be specified to lower than 1  $\mu\text{m}$ , the mean hardness value can vary by as much as 20% between observers. Therefore VAMAS recommended that hardness values, especially microhardness, should not be used for materials specification purposes. A guideline for hardness measurement of advanced ceramics has been produced and is being considered for standardization by CEN and other international standards organizations. Similarly, VAMAS work on fracture toughness has led to advancement of the technical knowledge necessary for standardization and further work is in progress.

**A classification scheme for advanced ceramics** As a relatively new category of materials, advanced ceramics has no formal classification scheme with the exception of IEC 672 covering electrical insulators. Conflicting schemes can impede efficient functioning of international trade and markets in advanced ceramic materials and products. Accordingly VAMAS initiated an activity in TWA14 with the aim of developing the technical basis for a unified classification scheme which will enable:

- Unambiguous materials specification;
- Unique specification for entries in databases;
- A link between materials and application;
- Unambiguous collection of statistical information on trade and markets.

Based on the findings of an extensive international survey of producers and users of advanced ceramics, a matrix coding system with the following four key elements has been developed<sup>(20)</sup>:

- Application (A)
- Chemical character and product form (C)
- Processing (P)
- Property data (D)

Such a system is very flexible and can meet simple to fairly complex requirements. For example, if only chemical composition is of interest only one element of the matrix is required. If a breakdown of markets by materials composition and application is needed, then two elements are used whereas all the elements may be necessary for materials specification. Such a system can be used by industry in the design of databases, product promotion, inventory purposes and analysis of market trends. Governments can use the scheme to gather economic statistics or to handle trade matters. ASTM and CEN are using the results of VAMAS as a basis for new classification standards, and it is expected that ISO will also adopt the scheme in the future.

## LABORATORY ACCREDITATION

Industry increasingly recognizes that in today's demanding and competitive markets, good product



design and efficient manufacturing must be underpinned by properly authenticated measurement and testing to ensure quality, performance and reliability of products. Laboratory accreditation plays an important part in this context because it signifies recognition by a third party of a Laboratory's competence to perform test and evaluation in specific fields. It provides confidence in the overall quality of operation and competence of a laboratory (although it does not guarantee that an individual test result or data is correct or valid). Accordingly many countries have set up in recent years national accreditation schemes which avoids multiple assessment of testing laboratories and provides uniformity in accreditation criteria and rules. On the international scene an increasing number of agreements are being reached for mutual recognition of test results across national boundaries. Industrializing countries are also examining accreditation schemes because of the realization that it has an important role in developing industrial capability and international trade.

To perform tests consistently from one day to the next, accredited laboratories should use standardized methods whenever available. From a user's point of view, the same test carried out in different accredited laboratories, even across national boundaries, to a standard specification should produce similar results.

Certified Reference Materials (CRM) serve a very useful purpose for interlaboratory comparisons and proficiency checks and are regularly used by accredited laboratories. A CRM has been defined by ISO as a material or a substance, one or more physical or chemical properties of which are sufficiently well established to be used for the calibration of an apparatus or for the verification of a measurement method. The material is accompanied by, or traceable to, a certificate stating the property value of concern and is issued by a nationally or internationally recognized and competent organization. Although there are various sources of supply for CRMs, both in the developed and the developing countries, CRMs for advanced structural materials have limited availability.

### STRATEGY FOR THE FUTURE

Standards are of strategic importance for stimulating new applications necessary to realize the market potential of advanced materials with consequent economic benefits as well as improvements in health, safety and the environment. In this respect the traditional view that standards are only needed to establish the basis for fair trading after a market has been established must be discarded.

In the early stages of market development in a rapidly advancing technological area as materials, there is a strong demand for standardized terminology for effective technical communication and technology transfer. To support innovation and indeed technological

progress, reliable information about the behaviour, properties and performance is required to give users the necessary confidence for incorporating new materials into their products thereby providing competitive advantages. With standardized test methods both producers and users of materials can have a common basis for materials evaluation and increased confidence in the properties data.

Throughout the world much R&D is being carried out in the field of advanced materials and significant benefits can be derived if the standardization and R&D activities are well integrated. This may be achieved by establishing proper co-ordination and channels between R&D and standardization for which both government and industry need to act in a cohesive fashion.

Appropriate capability in materials evaluation technology has to be built with partnership between the public and the private sectors. Generally, government can be expected to take the lead in the development of an infrastructure that will adequately support a wide range of industrial test and measurement activities. Understandably, industry is reluctant to invest in the underpinning work on developments of techniques and methods which are generic and have wide applicability. On the other hand, testing and generation of data on specific materials or for specific applications have more immediate commercial value such that industry can be expected to bear the costs. However, even in the infrastructural area, very close consultation and collaboration with industry is essential to ensure that the results are directly relevant and applicable in industry.

Quite commonly, in the standards area, the materials supply industries tend to dominate whilst users are not adequately involved. This is perhaps inevitable as user industries concentrate more on short-term problems facing the business rather than long-term issues. However, it is essential that user industries become involved from an early stage in the standardization process so that the most appropriate and useful standards are developed to maximise benefits to industry.

Trade in materials and their products is international in character and so it is vital that specification, codes of practice and standards are developed on an international basis whenever possible. This is now widely accepted. As a result we have seen Europe, USA and Japan all putting greater emphasis on international standards to facilitate the removal of technical barriers to trade and to increase industrial efficiency.

In the developing countries consumers accept lower quality goods due to economic pressures. This necessarily means that the standards do not have to be as rigorous. However, as economic ambitions of these countries increase and markets are globalized, their industries will need to employ higher standards in order to become more competitive on an international scale. Hence, standards work cannot be merely left to others.

To have an effective voice in the international standardization forum, developing countries need to participate with a good technical base. It is interesting to note here that in the recently set up ISO committee TC 202 on Microbeam Analysis, China has the Secretariat; also in TC 206 on advanced ceramics, several less developed countries are participating members. Only from a position of strength will industrializing nations be able to command respect and influence standards in the international arena.

It will be evident from earlier discussions that testing and evaluation of advanced materials can be highly complex, so a sound technical base is required before effective standards can be developed. Often this means that much pre-standards research, validation of methods and intercomparisons amongst laboratories are needed. International collaboration in this type of pre-standards and pre-competitive research can be beneficial on two grounds:

- (i) Cost of this type of work, which is quite significant, can be shared between countries;
- (ii) Any standard developed as a result is likely to be readily accepted by all partners because of the common technical base.

Not surprisingly the G7 nations use VAMAS as a mechanism for cost-shared action in pre-standards research on advanced materials. There is certainly scope for developing countries/regions to use similar models.

Good co-operation amongst developed countries could ensure that the leading laboratories including the facilities and expertise could be used to tackle priority problems in testing and evaluation in a way that brings considerable synergistic benefits. Co-operation could also be effective in developing certified reference materials.

Industrial design and manufacturing require increasingly sophisticated and high pedigree data in a form that can be integrated readily with advanced IT systems. Although many databases of materials properties are available throughout the world, their scope and quality vary widely and, not surprisingly, there is a strong demand for good quality data for advanced materials. The cost of producing and evaluating data can be very high, so data have commercial value. Nevertheless, there is scope for international co-operation in developing standards and accreditation procedures for databases which should enhance data utilization. Developing countries could benefit significantly from cost-sharing and co-operation in building databases of common interest.

Many of the developing countries have significant skills and expertise in materials characterization and testing. UNIDO have carried out consultations with several countries about their perception of the current

situation and future needs in this important area. Generally, the effort and resources are dispersed in these countries and it was commonly acknowledged that improved co-ordination and co-operation across national boundaries would be highly beneficial. UNIDO are exploring mechanisms for establishing a suitable framework that would enable improvements in technological capabilities of individual countries through co-operative work and at the same time enhance links with the activities of industrialized nations. Training, sharing of facilities, awareness of developments in testing and evaluation as well as establishment of information networks for standards, could be an integral part of the overall plan in this context.

## REFERENCES

- 1 "Emerging Technologies: A Survey of Technical and Economic Opportunities", Technology Administration, US Department of Commerce, (1990).
- 2 "Advanced Materials and Processing: The Federal Programme in Materials Science and Technology", A Report by FCCSET Committee on Industry and Technology to Supplement the President's FY93 Budget, (1992).
- 3 Japanese Industrial Standards Committee Report, "Standardization of Advanced Materials - Summary Report of the Special Committee on Standardization of Advanced Materials. JISC, Agency of Science & Technology, Japan, (1988).
- 4 H. Czichos, R. Helms and J. Lexow: Industrial and Materials Technologies: Research and Development, Trends and Needs, ISBN 3-894, 29-105-2, (1991).
- 5 B. E. Read, G. D. Dean and P. E. Tomlins, "Effects of Physical Ageing of Creep in Polypropylene, Polymer, Vol. 29, 2152, (1988).
- 6 ISO-DIS 11403/1, "Plastics - Acquisition and Presentation of Comparable Multipoint Data - Part 1, Mechanical Properties, (1992).
- 7 G. B. Thomas & R. K. Varma, EUR 14105EN (European Commission Report), "Evaluation of low cycle fatigue test data in the BCR/VAMAS intercomparison programme", Commission of the European Communities, Brussels, (1993).
- 8 F. A. Kandil and B. F. Dyson, "The influence of load misalignment during uniaxial low-cycle fatigue testing", I Modelling: "Fatigue Fract Eng Mat Struct, Vol. 16, No. 5, p. 509-27, (1993). II Applications: *ibid.*, pp. 529-37.

- 9 S. J. Bull, "Surface and Coatings Technology", 50, p. 25-32, (1991).
- 10 ISO, "A Vision for the Future", ISBN 92-67-10154-4, (1990).
- 11 CEN, "Standards for Access to the European Market": The Technical Programme, ISBN 92-9097-192-4, (1993).
- 12 J. J. Kübler, R. Morrell, "European Standardisation Activities for Technical Ceramics", ASME publication 93-GT-158, (1993).
- 13 A. D. Mercer, "ISO and Corrosion Standards" in "Corrosion Standards", pp. 14-22, published by the Institute of Materials, UK, ISBN 0-901716-09X, (1991).
- 14 D. E. Landlot, *ibid.* pp. 28-41, (1991).
- 15 G. C. Smith & M. P. Seah, "Surface and Interface Analysis", 16, 144-148, (1990).
- 16 M. P. Seah & G. C. Smith, "Surface Interface Analysis", 15, 751, (1990).
- 17 W. A. Dench, L. B. Hazell, M. P. Seah, "Surface & Interface Analysis", 13, p. 63-122, (1988).
- 18 VAMAS Bulletin No. 16, Published by NPL, ISSN 1016-2178, (1993).
- 19 R. Morrell, D. M. Butterfield & D. J. Clinton, Euroceramics, 3, 3.339-3.345, (1990).
- 20 VAMAS Report No. 15 - "Classification of Advanced Ceramics", ISSN 1016-2186, (1993).

Figure 1

Long-term prediction of creep in polypropylene from short-term results

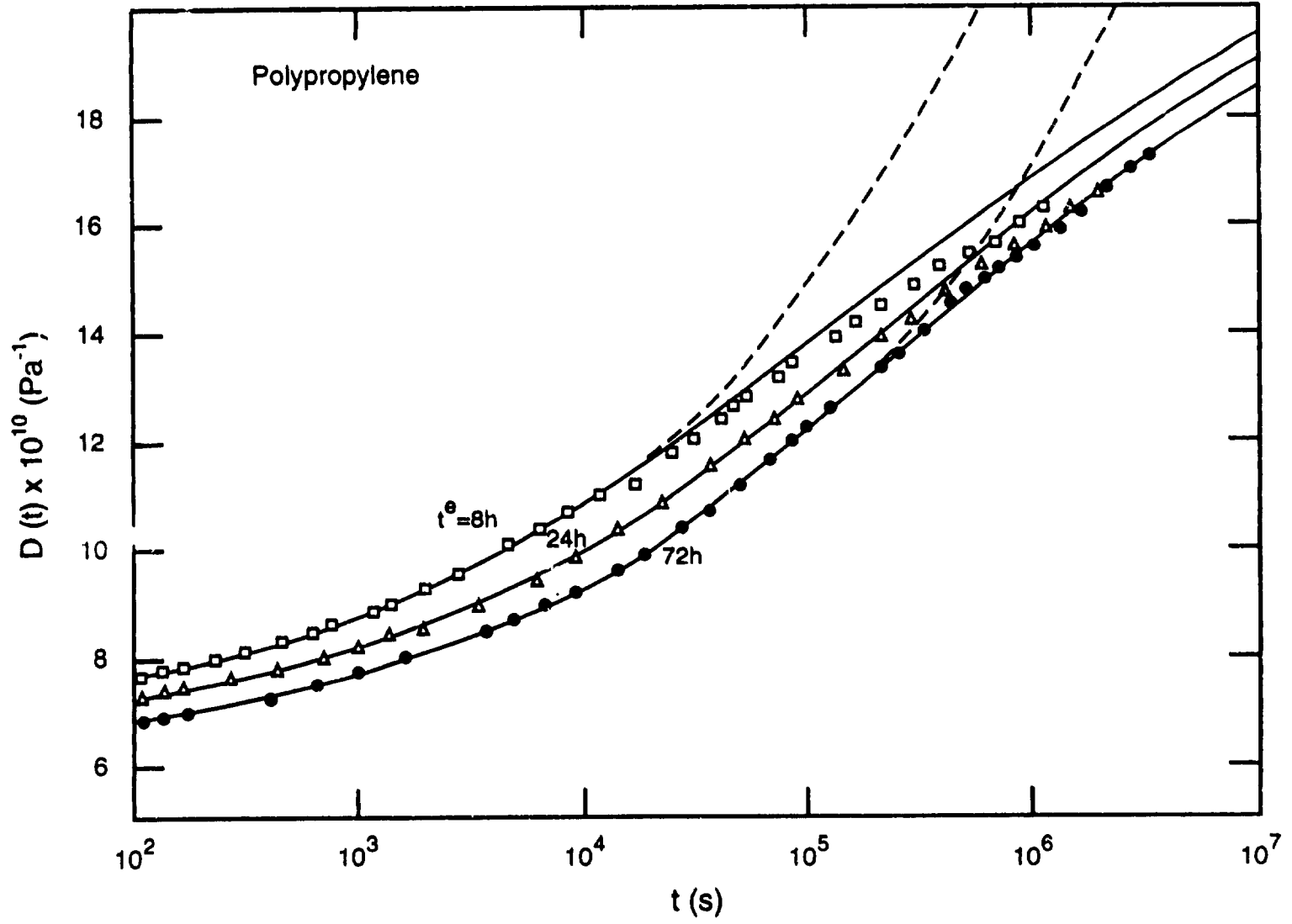


Figure 2

Results of an intercomparison exercise on the measurement of low cycle fatigue

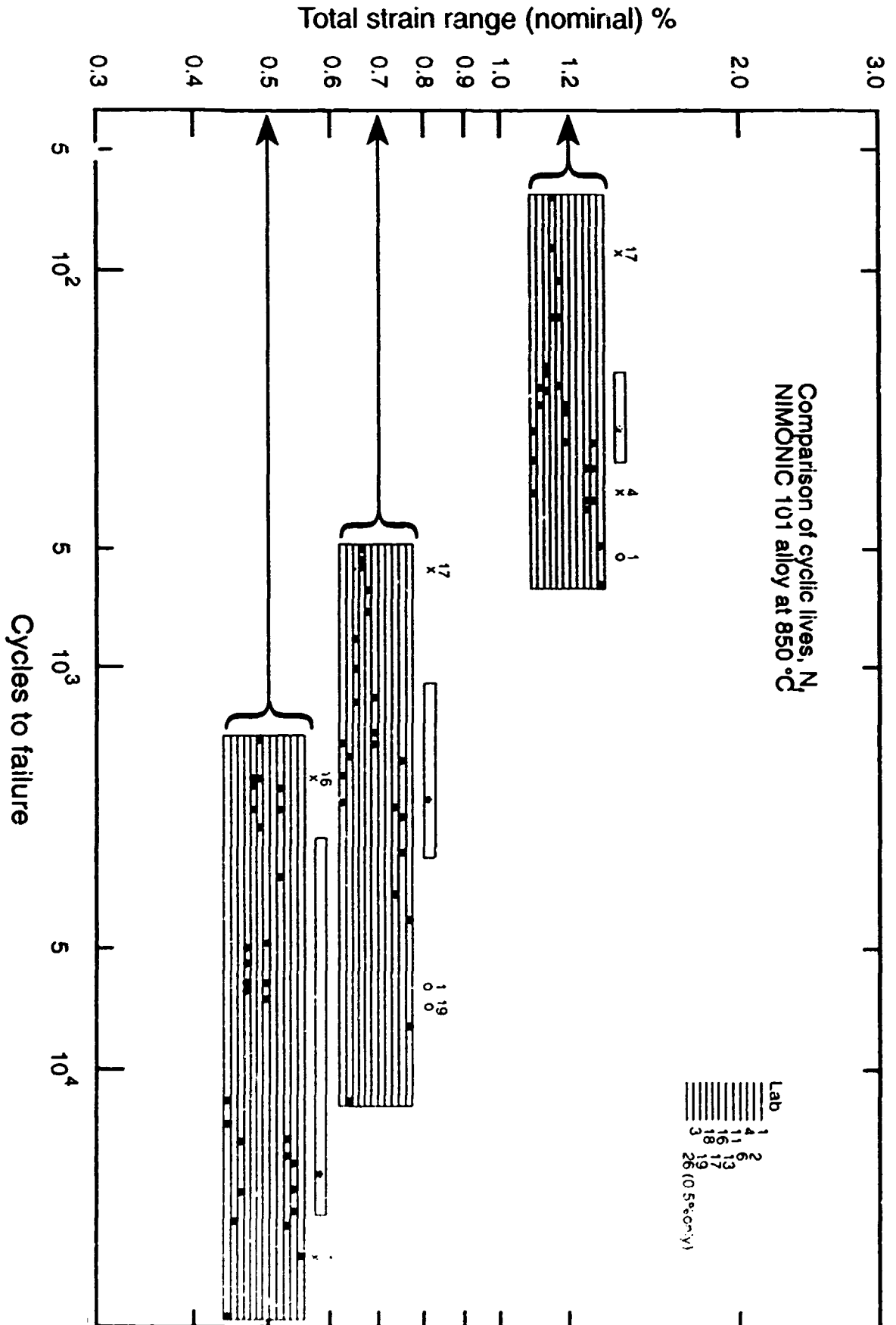


Figure 3

Comparison of a model predicting testpiece bending in LCF and experimental results

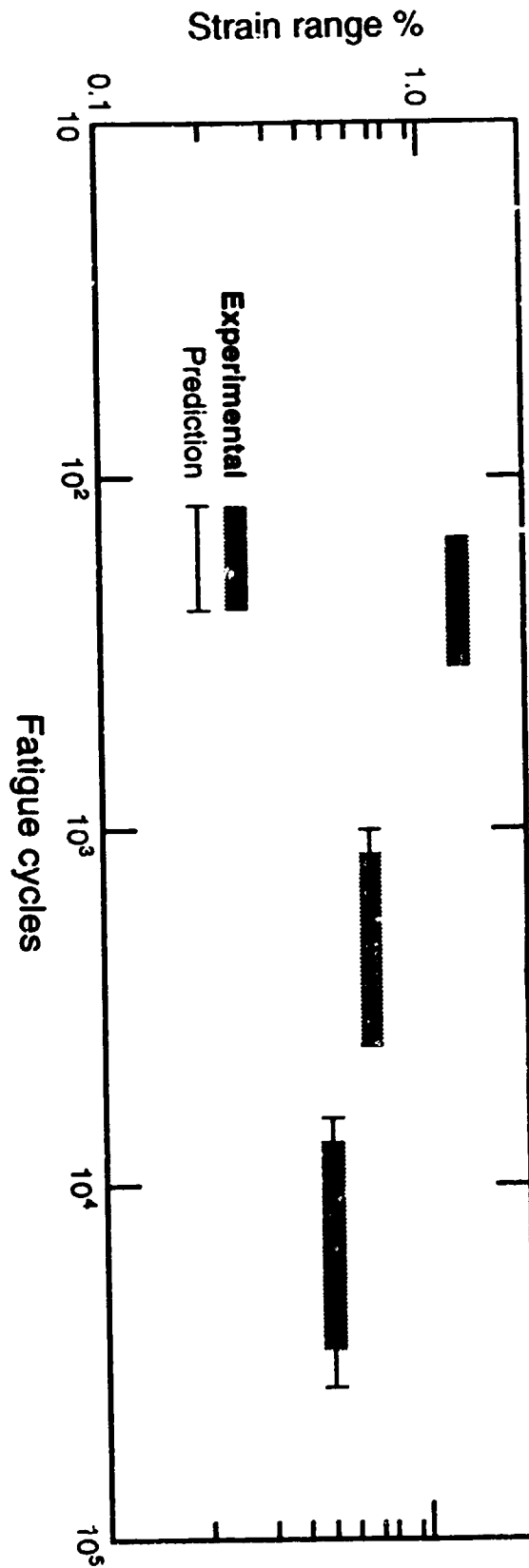


Figure 4

Current status and importance of various property measurements for engineering coatings

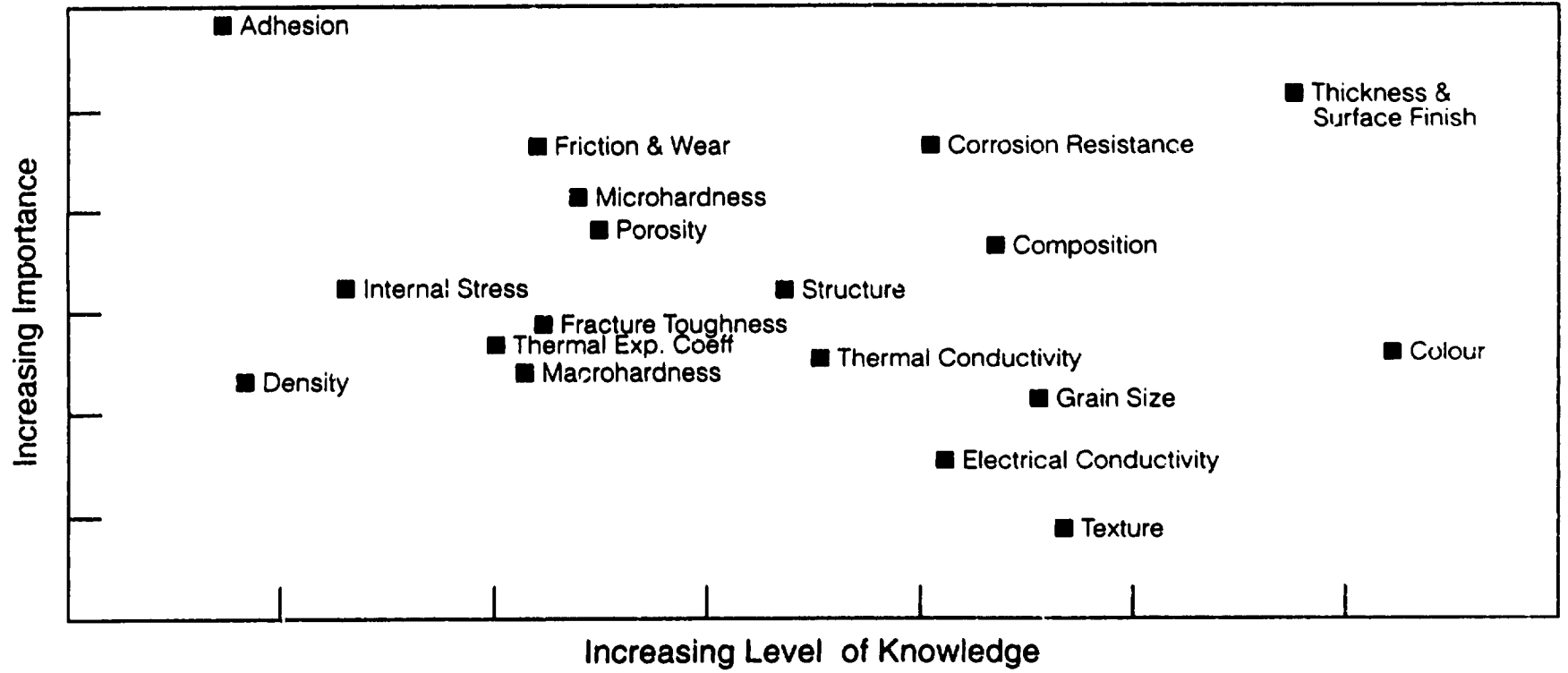


Figure 5

Use of Material data in process models and production

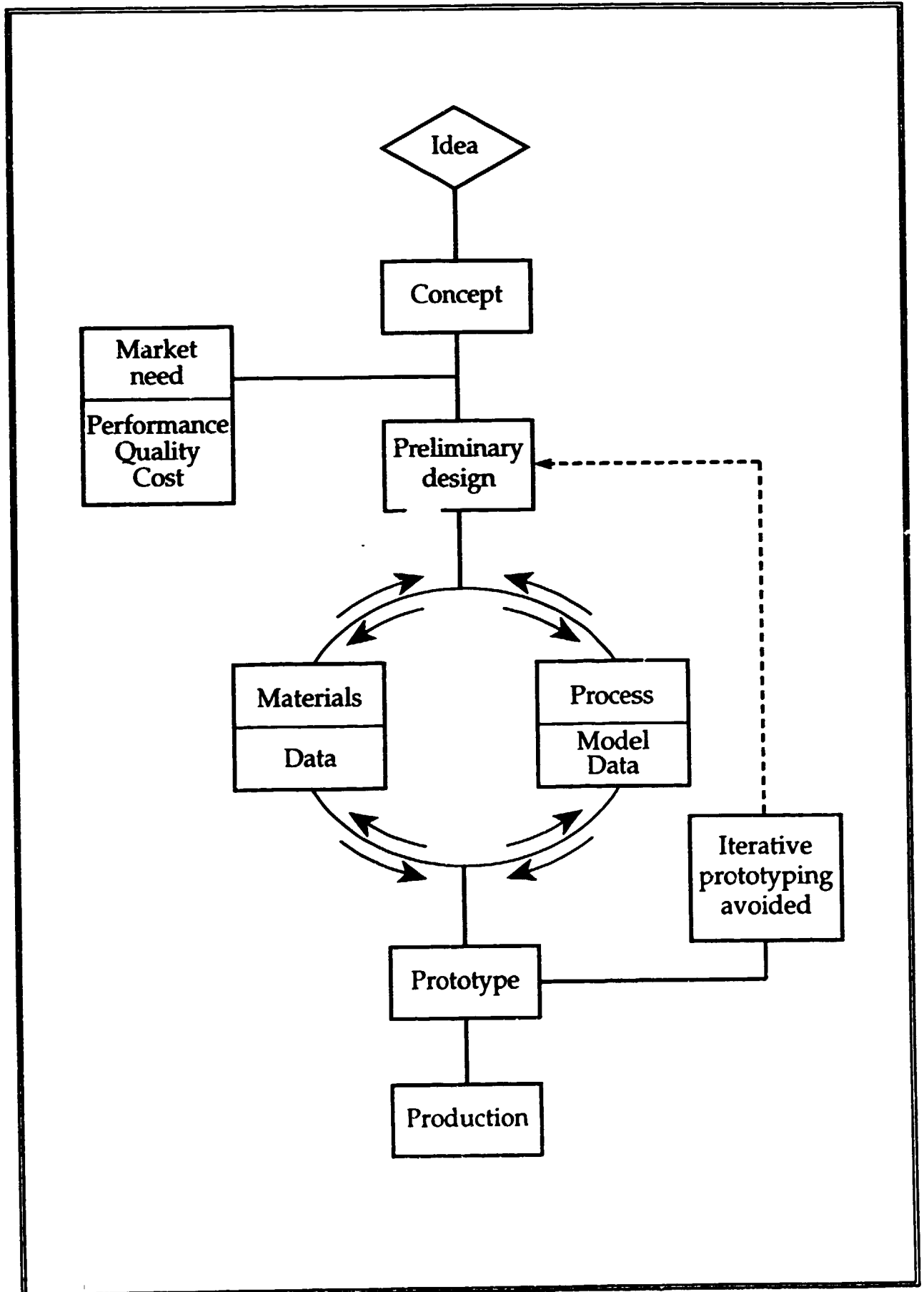
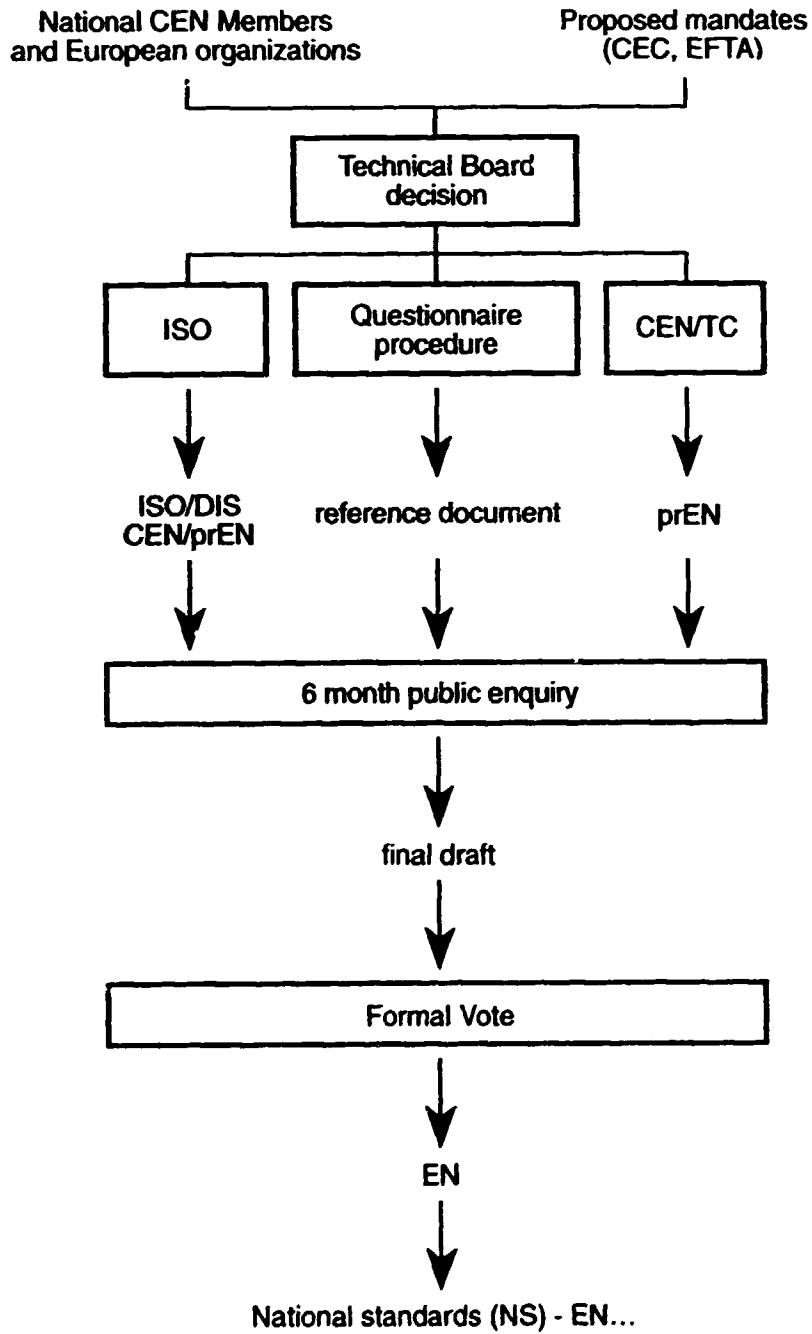




Figure 6

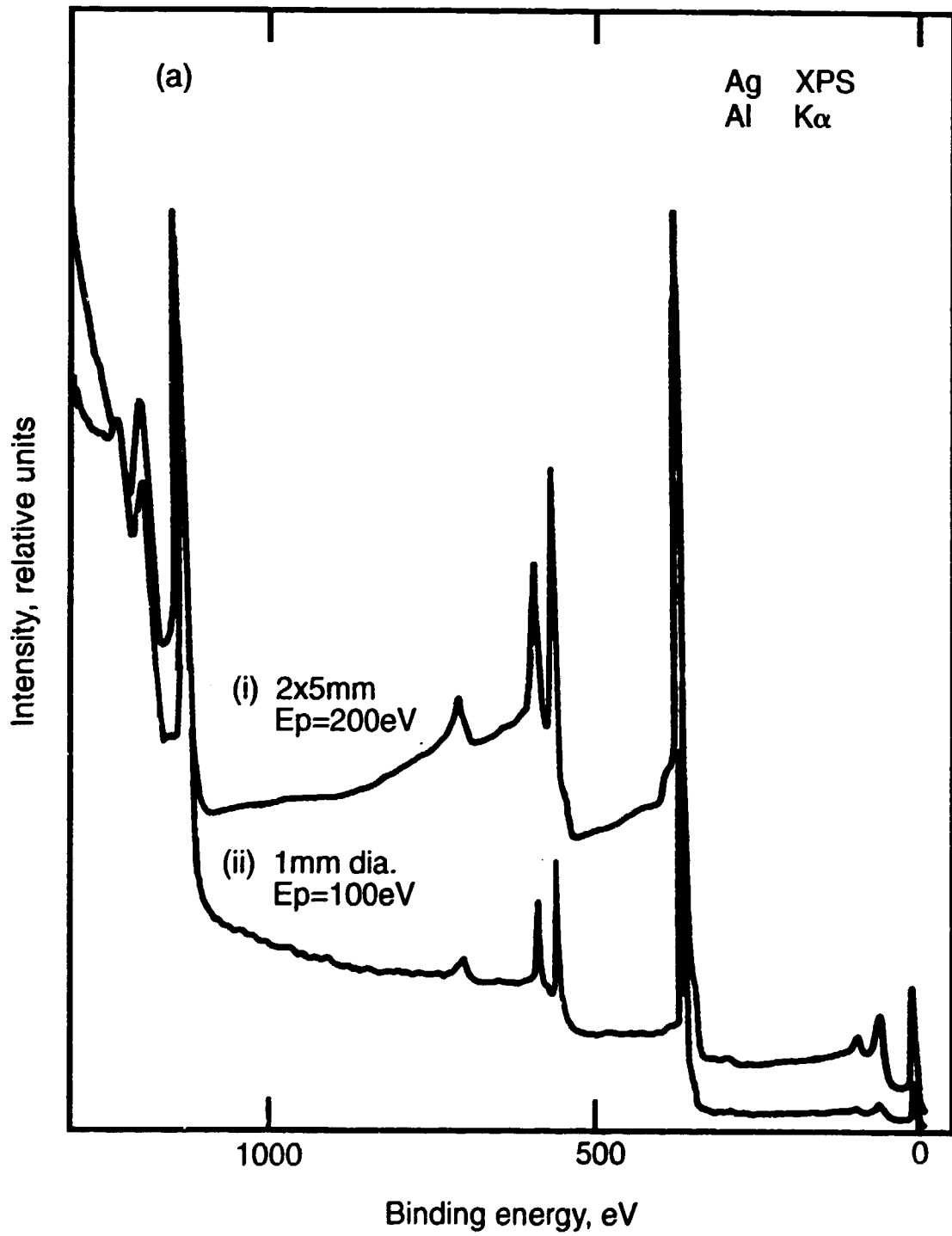
Key stages of standards development in CEN



Development of CEN standards

Figure 7

MG XPS spectra of Silver taken for two different analytical areas and pass energies



**Annex I**

**ISO Technical Committees in the Materials Field**

<b>ISO Technical Committees in the Materials Field</b>	
TC 33	Refractories
TC 35	Paints and Varnishes
TC 38	Cinematography
TC 45	Rubber and Rubber Products
TC 47	Chemistry
TC 61	Plastics
TC 76	Transfusion, Infusion and Injection Equipment for Medical Use
TC 79	Light Metals and their Alloys
TC 84	Syringes for Medical Use and Needles for Injections
TC 106	Dentistry
TC 119	Powder Metallurgy
TC 150	Implants for Surgery
TC 156	Corrosion of Metals and Alloys
TC 164	Mechanical Testing of Materials
TC 173	Technical Systems and Aids for Disabled or Handicapped Persons
TC 189	Ceramic Tile
TC 194	Biological Evaluation of Medical and Dental Materials and Devices
TC 201	Surface Chemical Analysis
TC 202	Microbeam Analysis
TC 256	Advanced Technical Ceramics

CEN/TC (or other)	Title	EN,ENV,HD,CR	Comments
<b>Metallic - ferrous, general</b>			
ECISS/TC 1A	Mechanical and physical tests	EN 10002-1, EN 10002-2, EN 10002-5, EN 10045-1, EN 10045-2	Testing methods for metallic materials
ECISS/TC 5		EN 10001	Definition, classification, conventional designation of pig iron and ferro alloys
ECISS/TC 6A		EN 10020	Terminology
ECISS/TC 6B		EN 10079	Terminology
ECISS/TC 7	Conventional designation of steel	EN 10027-1, EN 10027-2	Definition of steel names and steel numbers
ECISS/TC 9	Technical conditions of delivery and quality control	EN 10204	General technical delivery conditions for steel products - Inspection documents for metallic materials - Conformity assessment for steel products
ECISS/TC 20	Methods of chemical analysis	EN 10036, EN 10071, EN 10136 EN 10177, EN 10178, EN 10179, EN 10181, EN 10184, EN 10188, EN 10200, EN 24159, EN 24829-1, EN 24829-2, EN 24934, EN 24935, EN 24937, EN 24938, EN 24943, EN 24946, EN 24947, EN 29658	Defining methods of chemical analysis for iron and steel
ECISS/TC 21	Vocabulary of heat treatment terms		Terminology
<b>Metallic - ferrous, products</b>			
ECISS/TC 11	Sections - dimensions and tolerances		Definition of dimensions and tolerances for structural steel sections and bars
ECISS/TC 12		EN 10029, EN 10051	Definition of dimensions and tolerances for steel flat products for structural and pressure applications

CENTC (or other)	Title	EN,ENV,HD,CR	Comments
ECISS/TC 13	Flat products for cold working - Qualities, dimensions, tolerances and specific tests	EN 10130, EN 10131	Definitions of qualities, dimensions, tolerances and specific tests for steel flat products for cold working
ECISS/TC 15	Wire-rod - Qualities, dimensions, tolerances and specific tests		Definition of qualities, dimensions, tolerances and specific tests for steel rods for drawing or cold rolling
ECISS/TC 27	Surface coated flat products - Qualities, dimensions, tolerances and specific tests	EN 10142, EN 10143, EN 10147	Definition of qualities, dimensions, tolerances and specific tests for coated flat products
ECISS/TC 28	Steel forgings		Technical delivery requirements for all types of steel forgings
ECISS/TC 29	Steel tubes and fittings for steel tubes		Technical delivery requirements for steel tubes and fittings for steel tubes for all applications
ECISS/TC 30	Steel wires		Technical delivery requirements for steel wire and wire products
TCISS/TC 31	Steel castings		Technical delivery requirements for all types of steel castings
<b>Steels - other uses</b>			
ECISS/TC 10	Structural steels - Qualities	EN 10025, EN 10163-1, EN 101632-2, EN 10163-3	Definition of qualities of steels for structural applications
ECISS/TC 19	Concrete reinforcing steel - Qualities, dimensions and tolerances		Definition of qualities, dimensions, tolerances and specific tests for concrete reinforcing and prestressing steels
ECISS/TC 22	Steels for pressure purposes - Qualities	EN 10028-1, EN 10028-2, EN 10028-3, EN 10207, ENV 22605-1, ENV 22605-2, ENV 22605-3	Technical delivery requirements for steel bars and flat products
ECISS/TC 23	Steels for heat treatment, alloy steels and free-cutting steels - Qualities	EN 10083-1, EN 10083-2	Technical delivery requirements
ECISS/TC 24	Electrical steel and strip qualities - Qualities, dimensions, tolerances and specific tests		Definition of qualities, dimensions, tolerances and specific tests for electrical steels

CEN/TC (or other)	Title	EN,ENV,HD,CR	Comments
ECISS/TC 26	Tinplate and blackplate - Qualities, dimensions, tolerances and specific tests	EN 10202, EN 10203, EN 10205	Definition of qualities, dimensions, tolerances and specific tests for steel products (<0.5 mm thick) intended for packaging
<b>Metallic - non ferrous</b>			
CEN/TC 132	Aluminium and aluminium alloys	EN 23134-1, EN 23134-2, EN 23134-3, EN 23134-4	Product standards - Wrought and unwrought products
CEN/TC 133	Copper and copper alloys		Copper and copper alloys for general, electronic machining, building, electrical purposes; for heat exchangers, refrigeration etc
CEN/TC 209	Zinc and zinc alloys		Primary zinc and zinc alloys - Flat rolled products - Testing methods
CEN/TC 220	Tin and tin alloys		Pewter, pewterware and ingot tin - Methods of analysis
CEN/TC 306	Lead and lead alloys and oxides (provisional title)		Lead, alloys and oxides in unwrought or finished products and oxides. Committee created in 1992
CEN/CS M14		EN 26352, EN 26501, EN 27520, EN 27526, EN 27527, EN 28049, EN 28050, EN 28343	Chemical analysis
<b>Non-metallic</b>			
CEN/TC 66		EN 59, EN 60, EN 61, EN 62, EN 63	Tests for glass reinforced plastics. Committee disbanded in 1987
CEN/TC 172	Pulp, paper and board	EN 20216	Work mainly based on ISO work
CEN/TC 184	Advanced technical ceramics	ENV 820-2, ENV 658-3, ENV 658-4, ENV 658-5, ENV 658-6, ENV 820-2	Classification, terminology, sampling, test methods
CEN/TC 187	Refractory products and materials		Analysis and test methods

CEN/TC (or other)	Title	EN,ENV,HD,CR	Comments
CEN/TC 248	Textiles and textile products	EN 20104-A01, EN 20105-A02 EN 20105-A03, EN 20105-B02 EN 20105-C01, EN 20105-C02 EN 20105-C03, EN20105-C04, EN 20105-C05, EN 20105-C06 EN 20139, EN 20811, EN 22313 EN 2490, EN 29073-1, EN 29073-2, EN 29073-3, EN 29073-4, EN 29092	Mainly based on ISO work
CEN/TC 249	Plastics		Specifications and test methods
CEN/TC 289	Test methods for leather (provisional title)		Committee created in 1991
<b>Others</b>			
CEN/TC 138	Non-destructive testing	EN 473, EN 25580, EN 27963	Terminology, equipments, principles, methods, acoustics, ionization, liquid penetrant, magnetic, optical, leakage - Testing, qualification of NDT personnel. Part of the programme of work linked to the directive for simple pressure vessels
CEN/TC 190	Foundry technology		Technical delivery conditions, material specifications and testing methods for casting
CEN/TC 262	Protection of metallic materials against corrosion		Terminology, methods of test and evaluation of corrosion probability - Performance standards for corrosion protection by coatings, electro chemical protection, inhibitors. Partly based on ISO work
CEN/CS M11			Methods of test, chemical analysis. Based on ISO work

EN = European standard    ENV = European prestandard    HD = Harmonised Document    CR = CEN Report

	Standardization Achievable in a Short Period of Time	Standardization Requiring a Somewhat Longer Period of Time	Standardization Requiring a Long Period of Time	Totals
Standardization Especially Necessary	Coefficient of Thermal Expansion Tensile Strength (Room Temperature) Fracture Toughness (Room Temperature) Thermal Shock Resistance Hardness Sampling and Sample Preparation Methods Oxidation Resistance (Room Temperature) Particle Size Distribution (0.1 $\mu\text{m}$ or greater) Particle Absolute Specific Gravity Particle Tap Density Filling Characteristics Creep Strength Sintering Coefficient of Contraction Electromechanics Binding Coefficient Piezoelectric Stress Coefficient Transmission Factor Chemical Component Analysis Cutting Characteristics Thermal Conductivity	Thermal Conductivity Tensile Strength (High Temp) Fracture Toughness (High Temp) Static Fatigue (Creep) Mechanical Shock Resistance Friction Resistance Non-Destructive Inspections Non-Metallic Impurities Analytical Methods Corrosion Resistance (acid, alkali, salt solution) Maximum Particle Diameter Particle Shape (Aspect Ratio) Fluid Properties (Angle of Repose) Particle Diameter (Powder, Organizer Structure) Granular Body Properties Adhesion and Bonding Properties Components Shock Resistance Critical Temperature ( $T_c$ ) Critical Current Density ( $J_c$ ) Critical Magnetic Field ( $H_c$ ) Meissner Effect Absorption Coefficient Existing State Analysis Crystal Phase and Crystalline Characteristics Fluidity Modulus of Elasticity Poisson's Ratio	Design Standards Assurance Testing Particle Size Distribution (0.1 $\mu\text{m}$ or less) Secondary Particle and Aggregation Properties Powder Forming Characteristics Extrusion and Forming Properties Surface Phase Evaluation Grinding Characteristics Grindability Cutting Characteristics Friction Resistance Fatigue Crystal Volume Determination Form Determination Shapeability (Forming Characteristics) Granular Body Particle Size Granular Body Density Granular Body Strength Sintering Characteristics Bending Strength Tensile Strength Compressive Strength Torsional Strength Shearing Strength Multiaxis Strength Thermal Shock Fatigue Erosion Resistance Bonding Strength	
	Total 47	Total 39	Total 38	124
Other needs	22	62	11	95
Totals	69	101	49	219

Standardization Needs on Advanced Ceramics in Japan

Annex III



Annex IV

Standards and Programme of CEN TC 184

Standards completed by TC 184	EN(V) No	Future standards programme (1993-95)
<b>GENERAL</b> 1 Classification	-	<b>GENERAL</b> 1 Terminology
<b>POWDERS</b> 2 Impurities in Al <sub>2</sub> O <sub>3</sub> 3 Impurities in BaTiO <sub>3</sub> 4 O <sub>2</sub> in non-oxides (thermal ext) 83 O <sub>2</sub> in non-oxides (XRF) 5 Particle size distribution 6 Specific area 7 Absolute density 84 Tap density 85 Untamped density 8 Compaction 9 Sintering curve	EN 725-1 725-2 725-3 725-4 EN 725-5 EN 725-6 EN 725-7 EN 725-8 EN 725-9 EN 725-10 725-11	<b>POWDERS</b> 2 Impurities in zirconia 3 Crystalline phases in zirconia 4 Impurities in silicon nitride 5 Flowability of powders
<b>MONOLITHIC CERAMICS</b> 10 Sampling and testing 11 Cracks by dye penetration 12 Density and porosity 13 Grain size 14 Surface roughness 15 Flexural strength 16 Elastic moduli 17 Sub-critical crack growth 18 Hardness 19 Flexural strength, high temp 20 Deformation 21 Thermal shock 22 Thermal expansion 23 Thermal diffusivity 24 Specific heat	- EN 623-1 EN 623-2 623-3 623-4 EN 843-1 843-2 843-3 843-4 820-1 820-2 820-3 EN 821-1 EN 821-2 821-3	<b>MONOLITHIC CERAMICS</b> 6 Statistical evaluation of fracture testing 7 Microstructural analysis 8 Oxidation testing, method 9 Flexural creep 10 Fractography, standard practice 11 Elastic moduli, high temp 12 Fracture toughness (preliminary) 13 Chem corrosion testing, laboratory procedures
<b>LONG-FIBRE COMPOSITES</b> 25 Tensile strength 26 Compressive strength 27 Flexural strength 28 Shear strength (Compression) 86 Shear strength (3-point) 87 Shear strength (double punch) 29 Thermal expansion 30 Thermal diffusivity 31 (Deleted) 32 Specific heat 33 Density	658-1 658-2 658-3 658-4 658-5 658-6 ...-1 ...-2 ...-3 ...	<b>LONG-FIBRE COMPOSITES</b> 14 Notations and symbols (under discussion) 15 Tensile properties (inert atmosphere, 2000 °C) 16 Tensile properties (air, 1700 °C) 17 Shear strength, in-plane 18 Interlaminar shear strength, high temp (inert atmosphere, 3-point) 19 Tensile properties of a dry fibre tow 20 Tensile strength, single filament at high temp 21 Flexural strength (inert atmosphere, 2000 °C) 22 Flexural strength (air, 1700 °C) 23 Thermal conductivity 24 Compression properties (neutral atmosphere, 2000 °C) 25 Compression properties, high temp (air) 26 Resistance to crack propagation 27 Elastic properties of NDT 28 Resistance to creep
<b>FIBRE PROPERTIES</b> 34 Size content 35 Linear mass 36 Filament diameter 37 Filament strength	...-1 ...-2 ...-3 ...-4	
<b>COATINGS</b> 69 Chemical comp (E/PMA) 70 Thickness (step and cap grinding) 88 Thickness (grinding) 71 Topography (SEM) 72 Porosity (metallography) 73 Adhesion (scratch)	...-4 ...-1 ...-2 ...-6 ...-5 ...-3	<b>COATINGS</b> 29 Chemical comp (GDOS) 30 Chemical comp (XRF) 31 Chemical comp (AAS) 32 Chemical comp (ESCA) 33 Coating hardness 34 Residual stress distribution

Annex V

Review of potential for test method harmonisation

[C=carbon fibre, G=glass fibre, A=all fibres, P=plastics, \*=drafts,  
SRM=SACMA, AITM=Airbus Industries, ACO=ACOTEG,  
CRAG=Composites Research Advisory Group (for Aerospace), UK

Test	Methods	Assessment
1 Longitudinal Tension (0°)	ISO 527(P), ISO 3268(G), EN 2561(C)*, EN 2747(G)*, ASTM D3039(F), JIS 7073(C), CRAG 300(F)	Normally straight tabbed specimen. Tab design and material different. 1 or 2 mm thick and between 10 and 20 mm wide. Different moduli measurement methods. Harmonisation started. ISO draft
2 Transverse Tension (90°)	EN 2597(C)*, ASTM D3039(F), JIS 7073(C), CRAG 301(F)	As above but 2 mm thick and 10-25 mm wide. Harmonisation started. ISO draft
3 Multidirectional Tension	ASTM D3039(F), CRAG 302(F), ACO/TP/14	Similar methods except that CRAG includes any ±θ. ISO 527 Part 4 may cover
4 Longitudinal Compression (0°)	ISO 8515(G), ISO 604(P), EN 2850(C)*, ASTM D3410(F), ASTM D695(M), CRAG 400(F)	Similar philosophy (except D695) but differences in gauge area and support jig. 6.35-12.5 mm wide and 2-4 mm wide. More difficult area to harmonise because of many support jigs. CEN new draft
5 Transverse Compression (90°)	ASTM D3410(F), ACO/TP/8	Both compression options as for Test (4). CEN new draft
6 Multidirectional Compression	CRAG 401(F), ACO/TP/14	Similar specimen geometries as Test (3)
7 Flexure	ISO 178(P), EN 63(G), EN 2562(C)*, EN 2746(G)*, ASTM D790(P), CRAG 200(F)	ASTM and JIS include 4 pt. Older standards (eg ASTM) have a range of thicknesses, later 2 mm but EN(G) is 3 mm. CEN new draft
8 Interlaminar Shear	ISO 4585(G), EN 2563(C)*, EN 2377(G)*, ASTM D2344(F), CRAG 100(F)	Normally 5/1 span/depth. ASTM range of thicknesses. EN(C) is 2 mm but EN(G) is 3 mm thick. CEN new draft. CRAG 2 mm
9 In-plane Shear	ASTM D3518(F), CRAG 101(F), ACO/TP/9, AITM 10002	Normally 2 mm thick. ASTM and CRAG 25 mm wide and ACO is 16 mm. AITM 25 mm x 1 mm. CEN preparing new draft
10 Open Hole Tension	CRAG 303(F), ACO/TP/12, SACMA SRM5(F), AITM 10007, DIN 65559	Same specimen width/hole diameter (6/1) for metric and non-metric versions except AITM 5/1. Hole diameter 6 or 6.35 mm (CRAG) has a range for both values
11 Holed Compression	CRAG 402(F), ACO/TP/11, SACMA SRM3(F), AITM 10008, DIN 65560	As above for tension
12 Compression After Impact	CRAG 403(F), DIN 65561 AITM 10010, SACMA(ASTM)(F)	Boeing test method (ASTM R-R) gaining acceptance. in DIN draft

Annex VI

VAMAS Technical Working Areas

Area No	Title	Materials under study	Chair
TWA1	Wear Test Methods	Alumina, Silicon nitride, AISI 52100 steel	USA, Germany
TWA2	Surface Chemical Analysis	Wide ranging reference materials, metallic and non-metallic	UK
TWA3	Ceramics	Alumina, zirconia-alumina	USA
TWA4	Polymer Blends	Polycarbonate/ Polyethylene blend, Orgalloy R-6000 commercial blend	Canada
TWA5	Polymer Composites	Glass and carbon fibre reinforced resins	France
TWA6	Superconducting and Cryogenic Structural Materials	Niobium-tin and niobium-titanium filaments, cryogenic steels	Japan
TWA7	Bioengineering Materials	Hydroxyapatite, alumina, zirconia	Japan
TWA8	Hot-Salt Corrosion Resistance	Rene 80 and IN738, Nickel-base superalloys, protective coatings	UK
TWA9 (completed)	Weld Characteristics	304 and 316 stainless steels	UK
TWA10	Material Databanks	Creep and fatigue data from low and high alloy steels	EC,USA
TWA11	Creep Crack Growth	Chromium/molybdenum/ vanadium ferritic steels	UK
TWA12	Efficient Test Procedures for Polymer Properties	Wide range of polymers	UK
TWA13	Low-cycle Fatigue	IN718 and Nimonic 101 nickel-base alloys, 316L and 9Cr/1Mo steel	EC
TWA14	The Technical Basis for a Unified Classification System	Engineering Ceramics	USA
TWA15	Metal-Matrix Composites	Discontinuous SiC reinforced aluminium	USA

**Annex VII**

**Proposed subcommittees of ISO/TC 201 on Surface Chemical Analysis**

- |          |  |
|----------|--|
| <b>1</b> | <b>Terminology</b>   |
| <b>2</b> | <b>General Procedures</b>  |
| <b>3</b> | <b>Data Management and Treatment</b>   |
| <b>4</b> | <b>Depth Profiling</b>   |
| <b>5</b> | <b>Auger Electron Spectroscopy (AES)</b>   |
| <b>6</b> | <b>Secondary Ion Mass Spectrometry (SIMS)</b><br><b>(includes Sputtered Neutral Mass Spectrometry and Fast Atom Bombardment Mass Spectrometry)</b> |
| <b>7</b> | <b>X-Ray Photoelectron Spectroscopy (XPS)</b>  |

**Annex VIII**

**Proposed areas of work to be considered by the subcommittees  
(shown in parentheses) of ISO/TC 201 on Surface Chemical Analysis**

- |          |   |
|----------|---|
| <b>A</b> | <b>Instrument Specifications (AES, GDOS, SIMS, TXRF and XPS)</b>  |
| <b>B</b> | <b>Instrument Operations (AES, GDOS, SIMS, TXRF, XPS, Depth Profiling, and General Procedures)</b>                            |
| <b>C</b> | <b>Specimen Preparation (General Procedures)</b>  |
| <b>D</b> | <b>Data Acquisition (AES, GDOS, SIMS, TXRF and Depth Profiling)</b>   |
| <b>E</b> | <b>Data Processing for Qualitative Analysis (AES, GDOS, SIMS, TXRF, XPS, and Data Management and Treatment)</b>               |
| <b>F</b> | <b>Data Processing for Quantitative Analysis (AES, GDOS, SIMS, TXRF, XPS, and Data Management and Treatment)</b>              |
| <b>G</b> | <b>Reporting Results (AES, GDOS, SIMS, TXRF, XPS, Depth Profiling, General Procedures, and Data Management and Treatment)</b> |
| <b>H</b> | <b>Terminology (Terminology)</b>  |
| <b>I</b> | <b>Reference Materials (General Procedures)</b>   |

**GDOS** Glow discharge optical spectroscopy

**TXRF** Total reflection x-ray fluorescence spectroscopy

## 2. INFORMATION ON TESTING TECHNIQUES AND EQUIPMENT

### Ultrasonic inspection detects hidden damage in composites

Advances in ultrasonic inspection methods allow high-resolution non-destructive examination of damage in multilayer fibre-reinforced materials.

Laminated fibre-reinforced composite materials offer excellent durability and high strength- and stiffness-to-weight ratios. For this reason, they often provide superior performance to metals in both primary and secondary structural applications. However, polymer-matrix composites are susceptible to interlaminar delaminations, fibre fractures, matrix cracking, fibre/matrix debonding, and other damage that can degrade structural performance. Such damage can occur during manufacture, assembly, and/or normal service, and may have an influence on usage and repair decisions. 1/

Because of this potential for damage, it is important to be able to non-destructively inspect and characterize damage in fibre-reinforced materials. Ultrasonics represents a state-of-the-art technique for performing such inspections. Recent advances in transducers, instrumentation, and scanning hardware have increased the effectiveness of this method of detecting and characterizing flaws.

### Fundamentals of ultrasonic inspection

Ultrasonic images are typically produced by mechanically scanning an ultrasonic transducer in a raster pattern over an area of a structure and then displaying the reflected or transmitted energy in a suitable format. The scan is usually performed in a water tank, or with the transducer in a "squitter" nozzle; the water provides a suitable coupling medium for transmitting the ultrasonic energy from the transducer into the test material. Ultrasonic images can be displayed in real time, or data can be digitized and stored for later analysis and display.

Images are conventionally displayed as either C-scans or B-scans. A C-scan is a plan-view image where a colour scale is generally used to display signal amplitude or depth information. A B-scan is an image of a cross-section at one particular location of interest with colour typically indicating signal amplitude.

Common ultrasonic testing modes include pulse-echo and through-transmission. In the pulse-echo mode, a single transducer serves as both the ultrasonic transmitter and receiver. The transducer emits a short ultrasonic pulse and "listens" for the echo. If the part has no flaws, echoes are returned only from the near and far surfaces. If a part has a defect, an additional defect echo is returned, which is received by the transducer as a

signal between the echoes produced by the part surfaces. The time of this echo is related to the depth of the defect. One problem with the pulse-echo method is that defects very near free surfaces are often "masked" by echoes from these surfaces.

In the through-transmission mode, two transducers are used, one on each side of the part. One transducer acts as the transmitter and the other as the receiver. Defects in the structure either block or attenuate the signal transmitted through the material. Thus, a low signal amplitude or total loss of signal is usually observed from regions containing internal flaws. Through-transmission is often superior to pulse-echo for detecting near surface flaws; however, no information about defect depth can be obtained with the through-transmission method.

Successful ultrasonic inspection of composite structures often involves using a combination of pulse-echo and through-transmission modes, and choosing proper transducer parameters to enable detection of relevant defects. There are often trade-offs in specifying transducer frequency and focal length for optimum flaw detection: if the transducer frequency is too low, the ultrasonic wavelength is too long to enable either detection or resolution of small defects. If the frequency is too high, then depth penetration of the ultrasonic beam is limited; also, sensitivity to non-relevant defects very near the surface may be too high.

Low transducer frequencies (0.5 to 5 MHz) are typically used for sections thicker than 13 mm (0.5 in), such as aircraft parts having honeycomb cores and composite skins. For parts of medium thickness (2.5 to 13 mm, 0.1 to 0.5 in), a medium-frequency transducer (5 to 20 MHz) is best for examining defects that are deeper than 0.5 mm (0.02 in). Near-surface defects can be characterized using higher frequency transducers. For very thin parts, 2.5 mm (0.1 in) or less, a very-high-frequency transducer (30 to 75 MHz) does an excellent job of completely characterizing the damage. A high-speed, all-digital waveform processing system, such as a 400 MHz Panametrics Multiscan system, has the versatility to handle all of these cases, both for pulse-echo and through-transmission testing modes.

### C-scan images reveal impact damage

Laminated composite materials are very susceptible to damage from relatively low energy impacts. 1-4 These impacts can occur during normal use, such as the impact of hail or runway debris on composite aircraft structures. Damage can also occur during manufacturing and assembly. For example, dropped tools are a common cause of impact damage in composites. Low-energy impact damage generally takes the form of internal matrix cracks and delaminations.

Often there is no external, visually evident indication of damage.

Ultrasonic inspection also is valuable for studying the exact nature of delaminations between the various layers. For this composite panel, it was found that the delaminations were roughly elliptical in shape, and that the major axis of any individual delamination was aligned with the fibre direction of the interfacial ply farthest from the impact site. This characteristic damage state is also commonly observed in impact-damaged laminates. 2-4/

#### B-scan imaging of cross-sections

An ultrasonic B-scan is useful for obtaining cross-sectional information non-destructively. Specifically, a B-scan image displays signal amplitude as a function of wave transit time and position. Since the depth of any indication in the material is directly proportional to the transit time of the echo associated with that indication, this type of plot produces a cross-sectional view of the internal state of the material.

#### Looking through shadows

Damage or flaws near the surface of a specimen, particularly large flaws or delaminations, may reflect nearly all of the ultrasonic energy and block flaws from view that are deeper in the composite material. This is referred to as hidden or shielded damage.

Multiscan systems have the capability of obtaining pulse-echo images from each side of a specimen simultaneously with a through-transmission image. This technique is a good solution to the shielded-damage problem when the specimen geometry permits testing ultrasonically from both sides. That is, the two pulse-echo images, one from each side, reduce the region of the specimen that is blocked by near-surface flaws.

#### Imaging variable-thickness sections

A C-scan image shows amplitude or time data received from a specific depth range in the material. If the limits of the depth range are set too near material interfaces, the image may be dominated by non-relevant interface echoes that mask defects. Because of this, conventional C-scan imaging has limited effectiveness when used to examine materials with non-constant thicknesses. However, B-scan slices produce clear images that show the thickness variations. Flaws may then be easily identified separately from the interface echoes of the part.

#### New tools for testing composites

Materials and processes for fabrication of composites have improved substantially in recent years. Such advances have enabled the use of composites in many applications where determining actual structural

properties and producing flaw-free structures are important quality assurance issues. Ultrasonic testing is a key inspection method for meeting these challenges.

It has been widely recognized that testing at conventional frequencies (1 through 15 MHz) is adequate for finding large flaws in laminated composites that are 6.4 mm (0.25 in) in thickness or greater. However, many recent studies have shown that testing at higher frequencies provides the researcher and quality engineer with important new tools.

Testing with transducer frequencies as high as 125 MHz is now possible because of recent improvements made in the performance of transducers and ultrasonic instrumentation, and also because of the availability of high frequency data acquisition systems. It is expected that the trend towards the use of higher transducer frequencies will continue, although the practical upper frequency will be limited by the ability to penetrate the material being tested.

It is also expected that the utilization of combined frequency testing will increase. In this technique, a low frequency transducer is used to penetrate deeper into the part and a high frequency transducer is used to detect and characterize defects nearer the surface. This kind of testing provides the ability to acquire data from an array of several transducers during a single scan, facilitating the combination and interpretation of test results.

The availability of faster computers and all digital data acquisition systems has simplified the acquisition and storage of complete waveforms. The B-scan images presented in this article are examples of this capability. It is also practical to store waveforms from scans over large areas of a part. Regions scanned in this manner may include critical structural areas, or areas where defects were found during conventional scanning. These waveform data sets can be archived for future reference, or they can be processed for detection, characterization, and enhancement of flaws. 5/

Finally, essentially the same information can be extracted from non-destructive as from destructive tests for impact-damaged laminates. Destructive testing is difficult, costly, time consuming and, very often, undesirable. Thus, ultrasonic inspection - with the capabilities of inspecting a part from both sides and providing combined information from interface-gated C-scans and selective B-scans - is often superior to destructive inspection for analysis of impact-damage sites in composite materials. In the future, this technique will likely be widely used as an alternative to destructive testing methods.

#### References

- 1/ "Assessment of Damage Tolerance in Composites", by E. Demuts, R. S. Whitehead and

R. B. Deo: *Composite Structures*, Vol. 4, Elsevier Applied Science Publishers, England, 1985, pp. 45-58.

2/ "The Influence of Lay-Up and Thickness on Composite Impact Damage and Compression Strength", by E. G. Guynn and T. K. O'Brien: *Proceedings of the 26th AIAA/ASME/ASCE/AHS Structures, Structural Dynamics and Materials Conference*, American Institute of Aeronautics and Astronautics, New York, N.Y., 1985, pp. 187-196.

3/ "Ultrasonic Imaging of Impact Damaged Composite Panels", by B. D. Davidson, et al.: *Acoustical Imaging*, Vol. 19, H. Ermert and H. P. Harjes (Eds.), Plenum Press, New York, N.Y., 1992, pp. 587-597.

4/ "Ultrasonic Inspection of Thin Walled Composite Tubes", by T. E. Michaels, T. M. Krafchak and B. D. Davidson: to appear in *Review of Progress in Quantitative Nondestructive Evaluation*, Vol. 12, D. O. Thompson and D. E. Chimenti (Eds.), 1993.

5/ "Ultrasonic Methods for Detection of Micro Porosity in Composite Materials", by J. E. Michaels, T. E. Michaels, and S. J. Jönsson: to appear in *Review of Progress in Quantitative Nondestructive Evaluation*, Vol. 12, D.O. Thompson and D.E. Chimenti (Eds.), 1993.

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#### Selecting the right ultrasonic system

The availability of fast, inexpensive computers and digital electronics has revolutionized the field of automated ultrasonic inspection. There are a number of PC-based imaging systems available that are within the budgets of most laboratories. However, there are numerous factors to consider when specifying and purchasing a system:

System performance is affected by: stiffness and precision of the scanner; bandwidth, noise level, and resolution of the ultrasonic instrumentation; characteristics of the data acquisition system; and software performance and features. A lower cost system may compromise these performance characteristics, which limits its capabilities and usefulness.

If the system will be used for a specific inspection problem, solicit vendor assistance. Most offer

application assistance to potential customers. Information from several vendors should be compared before making a decision.

Define the ultrasonic methods required to inspect the part before the system is purchased, and make sure that the selected system supports all of these needs. For example, if successful inspection of the part requires waveform acquisition and analysis at 400 MHz and the purchased system does not have these capabilities, then the application will not work.

Consider systems that can be upgraded with new components as inspection technology advances. Modular systems that are offered by progressive vendors usually provide the best assurance that your system will stay near the leading edge of technology.

Are there other application areas where the system can be used? Ultrasonic inspection technology is also applicable to metals and ceramics. Buying a system that is specialized for one area of expertise may limit its usefulness for other tasks. Sharing a system between several application areas may increase its cost-effectiveness to an organization. (Source: *Advanced Materials & Processes*, March 1993)

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#### Damage characterization of a metal matrix composite using standard ultrasonic techniques

Standard ultrasonic tests were performed on a metal matrix composite (MMC) between mechanical cycling in an attempt to detect the initiation and propagation of damage. The MMC was subjected to an isothermal 650° C modified fatigue test with an imposed hold time of 20,000 s at maximum stress. At the end of each corresponding cycle, two different types of ultrasonic methods - reflector plate and backscattering - were performed in an attempt to determine the degree of damage incurred by the specimen. Modulus values for the specimen were also gathered, using corresponding load-displacement data after each cycle. The goal was to determine whether standard ultrasonic techniques could be used to detect the initiation and propagation of damage in MMCs.

#### Introduction

As MMCs become a larger part of materials research and testing, a real need has arisen to assess the capability of current non-destructive evaluation (NDE) techniques to better characterize damage initiation and failure in these materials. To predict the life and ensure the quality of these composites, it is important that we learn more about the failure modes of these MMCs at elevated temperatures and high stress levels. Non-



destructive tracking of the initiation and propagation of damage in these composites would be of great benefit to researchers.

This work gathered ultrasonic and mechanical fatigue data on a unidirectional eight-ply SCS-6/Ti-24Al-11Nb MMC with a molybdenum crossweave after a 20,000 s hold time at maximum stress and under 650° C isothermal conditions. This cycling was applied until the material failed. This type of loading and unloading with an imposed hold time is typical of what an advanced, high-speed aerospace structure might experience during flight. Therefore, detecting damage and determining when this material has been degraded enough to become unsafe is an important factor in developing these materials.

Standard ultrasonic techniques were selected in an attempt to non-destructively detect damage initiation. Specifically, they were a reflector plate and a 45-degree shear-wave backscattering scan. In an attempt to detect fibre/matrix debonding, oxidation of the molybdenum crossweave, and matrix and/or fibre cracks known to occur as part of the damage process, these techniques were performed after each cycle until failure occurred. 1/ The specimen was expected to last four to five cycles. 2/ Thus, it was hoped that ultrasonic inspections between each cycle would reveal the amount of damage occurring in the specimen. This article discusses the results of these techniques.

#### Test procedure

The specimen tested was SCS-6/Ti-24Al-11Nb with a molybdenum crossweave. It was cut from an eight-ply unidirectional plate so that the fibre direction was along the loading axis. The specimen was rectangular in shape, measuring 111 by 6.4 by 2.0 mm. The specimen was tested on a low-frequency, pneumatically controlled fatigue testing system. Both ends of the specimen were hydraulically gripped with about 9,000 psi of pressure. Load-displacement data were acquired at room temperature using a maximum stress of 120 MPa. The displacement data were recorded using a high-temperature extensometer with quartz rods. The room temperature modulus value was calculated before each corresponding cycle. The test specimen was heated to isothermal conditions at 650° C using a four-zone quartz lamp system. The mechanical cycling, controlled by in-house software, consisted of loading the specimen to a maximum stress of 725 MPa, maintaining a 20,000 s hold time at this level, and unloading (see figure 1, page 38).

The loading and unloading phases each lasted 180 s. At the end of each cycle, two ultrasonic scans were performed to determine the degree of damage in the specimen.

A Caldata 5-axis scanning system was used with a Panametrics 5052 broadband pulser/receiver and a

LeCroy 200 MHz, 8-bit analog-to-digital converter to acquire the ultrasonic data. The in-house software for data acquisition and imaging allowed user-selectable software gating. For the reflector plate scans, the focused ultrasonic beam was propagated through the specimen. A glass plate 6.35 mm thick was used to reflect the ultrasound (signal) back to the transducer. An Ultran broadband 10 MHz transducer with a 50.8 mm focus was used. The ultrasound was focused on the front surface of the specimen (approximately 68  $\mu$ s away in time). The scan was set up to capture the through-transmission (two-pass) signal coming back from the reflector plate.

The next technique used was a 45-degree shear-wave backscattering scan. This technique causes the ultrasound to propagate through the specimen at a 45-degree angle. It was anticipated that this technique would detect any damage perpendicular to the surface, such as cracked fibres and matrix cracking. A 25 MHz broadband Ultran transducer with a 50.8 mm focus was used. A gate was placed to acquire any signal reflected from perpendicular defects (figure 2, page 38)).

#### Results

Figure 3 (see page 38) contains the results of the room temperature modulus values from the load-displacement data after every 20,000 s of testing (scatter of 5 per cent is typical for this data). The total life of the specimen was 165,000 s, which corresponds to a little over eight cycles.

#### Discussion

Judging by the modulus values in figure 3, there is no significant change in the stiffness of the specimen. Because the fibres in the specimen carry the majority of the loading, this is not unexpected. Once the fibres crack, the specimen will fail too rapidly for a slow, steady decrease in the modulus value to appear. As the molybdenum crossweave slowly oxidizes over time, it is likely that the fibres are directly exposed to environmental effects. As this occurs, they are more likely to degrade and crack. The fracture surface shows the presence of oxidized molybdenum weave at each fracture plane. In the backscattering scan, the molybdenum wire seems to have oxidized and reflected less ultrasound over time. By using this technique, a connection might be made between the amount of molybdenum weave that has oxidized and the life of the material.

To demonstrate the appropriate sensitivity for detecting fibre cracks, a 45-degree shear-wave backscatter scan was made of a single, fractured SCS-6 fibre embedded in a Ti6-4 matrix.

From the reflector plate scans there seems to be some matrix damage or cracking along the edges of the specimen. This is seen by a slight narrowing effect

shown in some regions. Though the failure location coincides with an area seen as narrowing by the C-scan, these effects are also seen elsewhere in the specimen.

Conclusion

As expected, the modulus value does not decline slowly over the life of the composite, but declines rapidly as the fibres suddenly crack and the load cannot be carried any longer - making it more difficult to detect and predict failure in this material. Monitoring modulus change as an indicator of damage is a standard testing procedure; however, it is not appropriate for these materials under similar test conditions. Predicting a time or location for failure in these specimens is somewhat difficult because of the complex nature of the composite itself. A small thinning effect, seen in the specimen over certain areas, is most likely due to damage propagated by the oxidized crossweave. However, it is difficult to use interpretations of this damage to predict a failure point in the specimen. The backscattering scan indicates the degradation of the molybdenum weave which occurs over time, and this could possibly provide some indication of material life. Although some correlation may exist between the time to failure in the specimen and the state of degradation in the

molybdenum weave, the exact correlation is unknown. In conclusion, the two standard ultrasonic techniques may not be sufficient to detect damage initiation and propagation that lead to failure for MMCs. Different methods must be developed to better detect and understand the damage failure characteristics of this material.

References

1. Talreja, R., "Fatigue of Composite Materials: Damage Mechanisms and Fatigue Life Diagrams", *Fatigue of Composite Materials*, pp. 25-38, 1987. Technomic Publishing Co., Lancaster, PA.
2. MacLellan, P., and D. Stubbs, "Effects of Hold Times at Maximum Stress on SCS-6/Ti-24Al-11Nb MMCs", presented at the AIAA 18th Annual Mini-Symposium on Aerospace Science and Technology, March 1992.
3. Kortyna, B. R., "High Cycle Fatigue Behavior of an SCS-6/Ti-24Al-11Nb Composite", M. S. thesis, University of Dayton, Dayton, OH, July 1990.

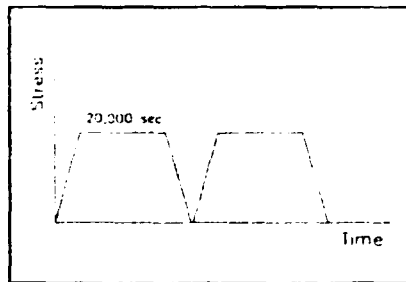


Figure 1 - Fatigue test loading profile.

45 DEGREE SHEAR WAVE, BACKSCATTERING

TRANSDUCER

22.5

8 PLY METAL MATRIX COMPOSITE

DEFECT

Cycle Number	Cumulative Test Duration (seconds)	Load-Displacement Modulus (GPa)
1	20,000	192
2	40,000	195
3	60,000	199
4	80,000	198
5	100,000	190
6	120,000	188
7	140,000	191
8	160,000	189
9	165,000	191

Figure 2 - Detection of defects using the 45-degree shear-wave backscatter technique.

Figure 3 - Elastic modulus data acquired before each fatigue cycle.

(Extracted from *Materials Evaluation*, October 1992, written by Patrick T. MacLellan, University of Dayton, 300 College Park Ave., Dayton, OH 45469)

### Ultrasonics for the non-destructive evaluation of materials

In cooperation with the Delft University of Technology TNO (TNO-P.O. Box 6070, 2 600 JA Delft, the Netherlands) is developing and applying high frequency (1-100 MHz) ultrasonic techniques for the non-destructive evaluation of materials, ranging from ceramics to composites and also human tissue.

Modern engineering materials such as metal alloys, technical ceramics and composites invariably rely on sophisticated and complicated processing in order to achieve optimum strength. High-frequency ultrasound can be used effectively for characterizing and validating these materials and the products made of them. In medical diagnostics one may discriminate between healthy and unhealthy tissue with the aid of high frequency ultrasound.

#### Flexible scanning system

Researchers of the TNO Institute of Applied Physics use an ultrasonic scanning system which has an open character, and which consists of several general purpose modules. A high-precision, computer-controlled scanning system together with various focused or unfocused transducers, allow for the flexible scanning of samples. Both reflection and transmission measurements can be performed. After amplification, the received signals are digitalized by a high speed oscilloscope, which passes the data onto the computer. Thus, both amplitude and phase are preserved which enables the application of parameter extraction techniques based on acoustic wave theory. Thanks to its open character, the ultrasonic scanning system can be used for investigating a wide variety of possible applications of acoustic microscopy e.g. in materials research and medical research.

#### Materials research

As ultrasound is a form of mechanical vibration, it provides information about the elastic properties of an object. For instance, the density, stiffness and hardness of an object can be determined on the basis of sound velocity measurements. For hardening layers, the layer depth can be determined from the frequency dependent behaviour of the velocity of surface waves. Surface waves are also used for inspecting ceramic materials for minute surface and subsurface cracks, which may result from, for instance, machining. When such measurements are repeated for many positions, these properties may be mapped into an image, giving good insight into the spatial dependency within the object.

Furthermore, ultrasound will interact - by reflection and diffraction - with internal structures within an object. Thanks to this phenomenon one can not only detect and image structural defects like cracks,

voids, inclusions and delaminations, but also measure such properties as porosity and grain size.

As for metals, TNO is using ultrasonic techniques for research on creep damage and hardening layers, while for ceramic materials research concentrates on the detection and imaging of defects like cracks, inclusions, voids and density gradients in bulk ceramics, coatings and bonds. In composite materials research the quantitative imaging of cracks and delaminations is the main goal.

#### Medical research

For dermatological diagnostics the Delft University of Technology has built a special scanner, which non-invasively produces cross-sectional images of the skin. Another application in the medical field is tissue characterization in coronary arteries. TNO carries out this work in close collaboration with the Erasmus University in Rotterdam. The ultimate goal is here to develop a catheter that enables cardiologists to accurately recanalize obstructed arteries, by discerning obstructions from healthy artery tissue with ultrasound. (Source: *Applied Research*, April 1990/30)

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#### Testing advanced materials at high temperatures

Innovative testing techniques and equipment are being developed to investigate the properties of advanced materials at temperatures approaching 2,200° C (4,000° F).

The performance of future automotive and aerospace propulsion systems and utility power plants can be significantly improved by using materials that withstand higher temperatures and stresses than those in current use. The National Aero-Space Plane and the Integrated High Performance Turbine Engine are just two examples of technological drivers for the development of new ceramics, composite materials, and metallic and intermetallic alloys that can withstand temperatures well in excess of the capabilities of current metallic alloys (-700° C, 1,300° F).

Facilities and techniques for testing advanced materials are being forced to keep pace with these new developments. To meet the need for very high-temperature testing, Southwest Research Institute (SwRI, Southwest Research Institute, 6220 Culebra Road, P.O. Drawer 28510, San Antonio, TX 78228-0510, USA; Tel.: 512/522-2338; Fax: 512/522-5122) is developing techniques and equipment to investigate the properties of monolithic and composite materials at temperatures approaching 2,200° C (4,000° F). These capabilities are complementary to the development of stress analysis and non-destructive testing techniques needed for comprehensive component design and assessment of service lifetimes.

SwRI is a non-profit research organization that provides a wide range of services, including materials development, materials property characterization, component design, and non-destructive testing development, for both the public and private sector. This article highlights several of the high-temperature macromechanics and micromechanics testing systems developed at SwRI.

The macromechanics systems provide data on the strength, fatigue, and tribological properties of materials mainly for engineering design of components, while the micromechanics facilities permit detailed experiments for determining and describing specific mechanisms of failure.

### Uniaxial and biaxial testing

One of the new systems developed at SwRI is capable of performing uniaxial monotonic or cyclic loading tests at temperatures up to 1,500° C (2,730° F). This system has been used to investigate the properties of composite materials over a range of temperatures, as a function of environment, under both static and cyclic loading.

Specimens are heated by using susceptors between the induction coil and the specimen. The coil produces an even heating zone of approximately 25 mm (1 in.) long. Temperature of the specimen, which is monitored with an optical pyrometer, is controlled by a thermocouple with feedback to the induction coil. The environment is controlled by continuously flowing gases through the quartz tube surrounding the specimen and susceptors. Either an oxidizing or non-oxidizing environment can be maintained depending on the gases supplied. Specimen displacements are measured with a capacitive extensometer having alumina "reach rods" and are recorded using a computer-based data acquisition system.

The system was used to test carbon matrix composites for the National Aero-Space Plane (NASP). The typical results are for carbon reinforced with a carbon-fibre cloth. Axial stresses of 70 to 90 per cent of the tensile strength were applied at an R ratio (minimum/maximum stress) of 0.1 at 1 Hz in both air and argon. Testing determined that a temperature increase from ambient to 1,100° C (2,010° F) decreased cyclic life by approximately a factor of 100; however, lifetime was not appreciably reduced by the presence of air during these short-term tests. A silicon carbide (SiC) conversion coating, approximately 0.25 mm (0.01 in.) thick, was applied to all test specimens to minimize oxidation, so the lack of environmental response indicated that the coating was able to withstand the combined effects of elevated temperature and repeated cycling.

Above 400° C (750° F), carbon oxidizes, which is manifest as pits or voids on the fibres. An examination

of fibres found on the fracture surface showed no evidence of oxidation, indicating that argon flow within the quartz tube was sufficient to keep the tests free of oxidation effects.

The unique feature of high-temperature testing systems capable of static or cyclic loading to 1,200° C (2,190° F) is that it can apply biaxial loads (tension or compression with torsion) to hollow specimens containing internal extensometers.

The system uses induction heating, either directly or through susceptors, to attain the high required sample temperatures. Loading and temperature changes can be controlled independently or synchronized, under computer control, to provide in-phase or out-of-phase strain and load-temperature cycling. Temperature is sensed by thermocouples attached to the specimen. Displacements, both axial and torsional, are measured by the biaxial extensometer within the tubular specimen. Data are sampled and stored by the same computer that controls specimen loading and heating.

### Microdisplacement measurements

A unique facility at SwRI is a scanning electron microscope (SEM) used to study the micromechanical and fracture properties of materials under load at elevated temperature. Specimens can be loaded in tension, either monotonically or cyclically, within the SEM at temperatures up to 850° C (1,560° F). Events preceding fracture are observed and photographically recorded using still photographs and video. Displacements resulting from the experiment can be visualized and measured from still photographs, allowing detailed study of the micromechanics of fracture in complex materials. Physically based models of the fracture process can be derived from development of improved material formulations and processes.

SEM is used instead of optical microscopy because the electron optics provide high resolution and good depth of field for observation of the sequence of events leading to fracture.

The first system to be constructed was a closed-loop, hydraulically actuated, cyclic loading stage for operation at ambient temperature. This was followed by construction of a similar stage for elevated-temperature service (fig. 4).<sup>1/</sup> Maximum load capacity of this system is 4,440 N (1,000 lbf), and the maximum cyclic rate is 8 Hz. Specimens up to several millimetres thick can be tested with this equipment.

Typically, the resolution available while using the loading stage at elevated temperature is about 0.1 µm, but less resolution (lower magnification) is often preferable for viewing a larger area of the specimen, such as the region surrounding a crack tip, so that both crack-opening displacement and deformation ahead of the crack tip can be measured.

Photographs are made at various loads and times during experiments using the SEM loading stage. These are compared using a stereo viewer to visualize the in-plane deformation. For example, in a fatigue-crack growth experiment, the deformation of interest might be that which occurred between minimum and maximum cyclic loads, so photographs made of the crack tip region under these conditions would be compared.

Likewise, if creep deformation is being investigated, photographs made at different times under constant load would be compared. In-plane deformation caused by changes in load or creep can be directly visualized using this technique, and deformation seen in the stereo viewer can be measured using aerial photogrammetry.

The technique developed at SwRI for measuring displacements and computing strains is called stereoimaging. <sup>2/</sup> True stereophotogrammetry uses two photographs of the same object taken at different locations. Stereoimaging, however, uses two images, intentionally distorted in relation to each other, taken at the same location.

New method: Recently, an image processing system capable of making improved photogrammetric measurements also was developed at SwRI. The DISMAP <sup>3/</sup> system (Measurement of microdisplacements by machine vision photogrammetry), is faster, cheaper, more accurate, and more reproducible than measurements made using conventional photogrammetry.

Photographs to be analysed are placed under the video cameras and aligned. The locations to be measured are designated and displayed on the video monitor. Displacements are then measured and displayed on both the monitor and (in numeric form) the graphics terminal.

The gradients of displacements are strains; therefore, three elements of the in-plane strain tensor can be computed from measured displacements, and from these, maximum and minimum principal strains, maximum shear strain, and effective strain can be found. Thus, it is possible to obtain seven values of strain at each of several hundred locations in a photographic field within a few minutes of conducting an experiment.

Use of the SEM loading stage with the DISMAP system has resulted in a detailed understanding of the micromechanics of deformation and fracture of high-temperature alloys and composites systems, and has improved considerably the understanding of high-temperature failure mechanisms. Measurements have also allowed theories and hypotheses to be tested and new models to be developed.

Case histories: The combined SEM loading stage and displacement measurement system developed at SwRI has been used for tensile deformation, fatigue-

crack initiation and growth, creep, creep-fatigue interaction, and fracture toughness testing of numerous metallic alloy, composite, and ceramic specimens.

For example, the tensile deformation of a glass-ceramic-matrix composite was studied at ambient and elevated temperatures. Silicon carbide fibres reinforcing the matrix were 20  $\mu\text{m}$  in diameter and spaced irregularly in the matrix. A photomicrograph of the specimen, at 800° C (1,470° F) and a load approximating that needed to fracture the matrix. The displacements measured for this condition are superimposed on the image. Strains in the matrix and in the fibres were determined from this analysis and the loads at which fibres debonded from the matrix were measured.

In another application, the fracture characteristics of titanium aluminides were studied by growing fatigue cracks in vacuum at 800° C (1,470° F). <sup>4/</sup> The microstructure of this material is a combination of equiaxed grains and a lamellar structure, with the crack tip approaching the boundary between the two. This strain distribution is unlike that predicted by theory.

Creep-crack growth also has been studied in a fine-grain glass-ceramic. The strain field was determined in the region ahead of the crack tip. Strain fields near the crack tip are not homogeneous and are not predicted by theory. <sup>5/</sup> A comparison of this strain distribution with that found for a creeping crack tip in AISI Type 304 stainless steel at 600° C (1,110° F), shows just how unusual the strain distributions are for this glass-ceramic.

#### Studying creep-induced damage

The primary high-temperature creep testing facility for ceramics and ceramic composites at SwRI is a dead-weight-loaded machine. Designed and built by the institute, the machine can be set up to apply loading in tension or compression. Heat is supplied by a furnace that uses refractory-metal elements capable of producing temperatures up to 2,300° C (4,170° F) in vacuum and 2,000° C (3,630° F) in inert gas. The load train has a self-aligning grip system to minimize bending moments in the specimen during tensile experiments. This high-temperature equipment is being used extensively to perform fundamental creep studies specifically aimed at investigating the mechanisms of creep damage development in a number of advanced ceramic materials.

The critical mechanism for creep failure in ceramics is the nucleation, growth, and coalescence of cavities on grain boundaries. <sup>6/</sup> These cavities nucleate because of the stresses that arise at grain boundary second-phase particles, ledges, and triple points that result from stochastic grain boundary sliding (GBS). <sup>7/</sup> Creep-induced damage is detected through the use of small-angle neutron scattering (SANS), <sup>6/</sup> a powerful technique that detects closed cavities in the interior of ceramic specimens. A large volume of material can be

assayed by the neutron beam, allowing measurements of cavity size, distribution, spacing, and morphology over a size range of approximately 1 to 1,000 nm. The SANS work was performed in collaboration with the National Center for Small-Angle Scattering Research at Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.

A number of measurements were made during these studies that contradicted current creep models. For example, it was found that the creep damage process was more dominated by cavity nucleation than by cavity growth, as is often assumed in models. Through analysis of the SANS data it was deduced that continuous nucleation of pores occurred during creep and that the apparent steady-state growth rate of individual cavities was nearly zero. This indicated that local stress buildup in grain boundaries was the driving force for cavities to nucleate, and when this stress relaxed through creep deformation, no further driving force was present for cavity growth. Also, SANS data showed that most cavities nucleated on grain boundary facets and not at triple points, as previously thought.

The results of this study promoted the development of a model to predict creep rupture times from the creep process in terms of cavity nucleation and growth, and stochastic GBS. 7/, 8/

GBS displacements and strains in aluminium oxide undergoing creep are being measured through use of the creep frame together with the DISMAP system. In the test, a cylindrical compression specimen, having a plane polished on the radial surface, was subjected to compressive creep at 30-minute intervals. At the end of each interval, a replica was taken of the planar surface to record microstructural changes. Optical micrographs were then taken from the same area as the replicas at each time interval. GBS displacements were measured from these photos using the DISMAP system.

In a specimen undergoing creep at 1,600° C (2,910° F) and 140 MPa (20 ksi), it was observed that the relative magnitude and direction of GBS varies from boundary to boundary.

Grain boundary widths measured by transmission electron microscopy (TEM) were used to compute boundary strains greater than 4,000 per cent at rates approaching 200/s. 9/ Contrary to prior belief, no relationship was found between the shear along the boundary and the corresponding grain boundary orientation to the compressive load axis. 10/ A large number of GBS measurements are being collected and will be combined with SANS cavitation measurements to develop a more comprehensive model describing the relationship between GBS and cavitation.

### Friction and wear testing

Improved performance of advanced heat engines, diesel engines, and gas compressors will require materials having superior friction and wear behaviour at elevated temperatures. To test materials for these applications under realistic conditions, SwRI has constructed specialized facilities for measuring friction and wear at temperatures up to 1,000° C (1,830° F).

One of these facilities, the reciprocating high-temperature friction and wear rig, allows bench testing of new materials for high-temperature tribological applications. This unique facility simulates the harsh elevated-temperature environment inside internal-combustion engines.

For applications in an uncooled internal-combustion engine, SwRI has produced self-lubricating ceramics exhibiting minimal wear at high temperature (600 to 900° C, 1,110 to 1,650° F) by modifying surface and bulk properties.

The surface-modified ceramic was produced by ion-implanting partially stabilized zirconia (PSZ) with titanium and nickel. When tested against Ni-Mo-bonded titanium carbide (TiC) at 800° C (1,470° F), engine liners made of the ion-implanted ceramic exhibited better friction and wear performance than conventional cast-iron baseline components. Ion-implanted PSZ exhibited coefficients of friction on the order of those observed in a lubricated metal system (=0.1). 11/

Bulk-material modifications were developed to ensure a continuous distribution of lubricant throughout the specimen. Specifically, silicon nitride ( $\text{Si}_3\text{N}_4$ ) was reinforced with TiC particulates and SiC whiskers to form a self-lubricating composite that exhibited little wear compared with monolithic  $\text{Si}_3\text{N}_4$  at 900° C (1,650° F). The  $\text{TiC}_p/\text{SiC}_w/\text{Si}_3\text{N}_4$  composite exhibits a coefficient of friction ( $\mu_f$ ) half that of the monolithic  $\text{Si}_3\text{N}_4$  under identical conditions. 12/ The mechanism for self-lubrication at elevated temperatures was found to be the formation of a lubricous oxide film containing titanium and silicon.

### Other high-temperature systems

Other specialized test systems also have been designed and constructed at SwRI to meet the needs for elevated-temperature experimentation. One of these, the split Hopkinson pressure bar, is designed to apply loads at rates between  $10^3/s$  and  $10^4/s$ . It can test specimens in compression from -160 to 2,300° C (-255 to 4,170° F). At ambient temperature, the system can be used with confining pressures up to 550 MPa (80 ksi).

Modifications to the current elevated-temperature fatigue testing system are planned so that tests up to 2,000° C (3,630° F) can be performed. Changes will include a water-cooled environmental chamber, and a new extensometer with coated zirconia composite reach rods.

Another fatigue testing system is being designed for service up to 1,600° C (2,910° F) in vacuum or other environments, with capability to observe and photograph the specimen at magnifications up to 800X. This facility will extend the ability to make micromechanics measurements beyond the current limit of 850° C (1,560° F).

### References

1. "A high temperature cyclic loading stage for the SEM", by A. Nagy, J. B. Campbell, and D. F. Davidson: *Rev. of Sci. Instrum.*, Vol. 5, 1984, pp. 778-782.
2. "Fatigue crack tip plastic strain by the stereomaging technique", by D. R. Williams, D. L. Davidson, and J. Lankford: *Experimental Mechanics*, Vol. 20, 1980, pp. 134-139.
3. "Measurements of microdisplacements by machine vision photogrammetry (DISMAP)", by E. A. Franke, D. J. Wenzel, and D. L. Davidson: *Rev. of Sci. Instrum.*, Vol. 62, 1991, pp. 1270-1279.
4. "Fatigue crack growth through the lamellar microstructure of an alloy based on TiAl at 25 and 800° C", by D. L. Davidson and J. B. Campbell: *Metall. Trans.* (in review).
5. "The micromechanics of creep crack growth in a glass-ceramic", by R. A. Page *et al.*: *J. Am. Ceram. Soc.*, Vol. 73, 1990, pp. 2977-2986.
6. "Creep cavitation in liquid-phase-sintered alumina", by R. A. Page *et al.*: *J. Am. Ceram. Soc.*, Vol. 70, 1981, pp. 137-145.
7. "Continuous creep cavity nucleation by stochastic grain boundary sliding", by K. S. Chan and R. A. Page: *J. Mat. Sci.*, Vol. 25, 1990, pp. 4622-4629.
8. "Stochastic creep cavitation in metals and ceramics: Part 2 - cavity coalescence", by K. S. Chan and R. A. Page: to be published in *Acta Metall.*
9. "Grain boundary sliding measurements in Al<sub>2</sub>O<sub>3</sub> by machine vision photogrammetry", by C. R. Blanchard and R. A. Page: *J. Mat. Sci.*, Vol. 26, 1991, pp. 3165-3170.
10. "Grain boundary sliding microdisplacement measurements during the creep of Al<sub>2</sub>O<sub>3</sub>", by C. R. Blanchard and R. A. Page: *J. Mat. Sci.*, Vol. 75, 1992, pp. 1612-1620.
11. "Friction and wear testing of ion beam modified ceramics for high-temperature adiabatic diesel engines", by W. Wei *et al.*: *Selection and Use of Wear test Systems for Ceramics*, C. S. Yust and R. G. Bayer (Eds.), American Society for Testing and Materials, Philadelphia, PA., 1988, pp. 74-87.
12. "Effect of SiC whiskers and TiC particulate additions on the friction and wear behaviour of Si<sub>3</sub>N<sub>4</sub>", by C. R. Blanchard and R. A. Page: *J. Am. Ceram. Soc.*, Vol. 73, 1990, pp. 3442-3452.

(Extracted from *Advanced Materials & Processes*, November 1992)

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### Accelerated fatigue testing of crankshafts

British Steel specialist (Shardlow Ltd. of Sheffield) recently came out with a resonant dwell fatigue test equipment. This offers accelerated testing with the added bonus of lower energy consumption.

With the resonant dwell method, heavy inertia weights are attached to the adjacent main bearings of the crankshaft which, in turn, is isolated from its supporting structure by elastic couplings. The resulting "tuning fork" is then excited at fixed amplitude and fixed acceleration by an external energy source until resonance is achieved.

At this point, the sinusoidal output waveform is the same as the input waveform, but with a phase shift of 90°. Also, at this frequency, the power required to sustain the test is minimized.

Resonance is maintained and the test run continued until the electronic feedback system registers a change in the resonant frequency of the crankshaft. This change occurs when the specimen suffers fatigue failure. Normally, a circumferential crack will be observed in the pin-bearing fillet radius. When the resonant frequency changes by a given amount from the original set value, the rig automatically shuts down and the number of cycles to failure is recorded.

Stress in the fillet radius is obtained by converting the input acceleration value to a specific strain. The necessary correlation is established prior to the test by a strain gauge analysis. Youngs Modulus and Poissons Ratio are employed to eliminate hoop stress and determine pure stress only in the line of bending.

Several crankshaft sections are tested in this way. The resulting data, stress at varying acceleration values,

are plotted against the number of oscillations to give the familiar SN curve.

Minimum fatigue life can be interpolated from the data to the strength of the material in pure tension. Fatigue strength distributions obtained by this method can then be compared with application requirements, represented by a similar distribution. The adequacy of the crankshaft strength can be judged from the area overlap of the two distributions.

The test procedure is relatively simple to conduct and the full analysis can be completed within 14 days. (Excerpt from *Steel Times*, March 1993)

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#### Non-destructive way to ID defects in silicon wafers

Applying a microscope and a new laser, Mitsui Mining & Smelting Co. Ltd. has developed a non-destructive method to find defects in intact silicon wafers.

In the new technique, a wafer is put to an imaging examination through the use of a microscope and a laser. This enables the observation of the various cross-sections of a wafer without having to cleave it. The system can be installed on a production line for continuous on-line inspection. (Excerpt from *American Metal Market*, 31 October 1990)

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#### Superbright X-rays to study materials

E. I. du Pont de Nemours & Co. Inc., Dow Chemical Co. and Northwestern University have entered into a research collaboration to use a "superbrilliant X-ray facility" now being constructed at Argonne National Laboratory to probe into materials in ways never before possible.

The companies are forming a team of scientists and engineers to operate one sector of the Advanced Photon Source (APS) at Argonne, which will produce the most powerful and focused X-rays available when it becomes operational in 1996.

The companies said each sector of the APS includes two X-ray beams "with unprecedented brilliance. The X-ray beams will be 10,000 times brighter than anything currently available and 10 billion times brighter than routine medical X-rays."

The APS will allow researchers to probe the atomic and molecular structure of advanced materials and develop critical production and application data.

It also will allow scientists and engineers "to design new varieties of materials as diverse as fibres,

engineering polymers, catalysts, construction materials, composites, ceramics and even superconductors". (Extracted from *American Metal Market*, 24 February 1993)

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#### New concept for rapid fatigue testing of welded joints

The application of ultrasonic vibrations in the area of manufacturing processes such as drilling, welding and soldering is well known. These applications are based on the high amplitude vibrations of the tool/workpiece, etc., at a frequency of (usually) around 20 kHz. What is perhaps little known (at least in India) is that ultrasonic vibrations can also be used to generate extremely high stresses (cyclic) within the body of a material leading ultimately to its failure. The uniqueness of the process lies in the fact that there is absolutely no external loading of the specimen, the stresses being, *per se*, generated by the ultrasonic vibrations themselves. It is necessary of course that the specimen conform to certain dimensions dictated by the wavelength of the sound in the material concerned. Such failure has indeed been found to occur within a few minutes of insonation (e.g. conventional fatigue which takes several hours to produce failure). Fatigue is of the high cyclic variety only but even so takes only a few minutes to be completed at these very high frequencies.

This concept can also be usefully deployed in evaluating the integrity of welded joints under fatigue. Furthermore it is possible (in principle at least) to concentrate the loading at specific locations in the sample - e.g. the joint itself or a little farther away as in the HAZ or the base metal. It is thus possible to make absolute comparisons of the strength of a joint with respect to other regions. There is no other facility commercially available which could make such a comparison. (Source: *Research and Consultancy of IIT Madras*, Vol. 12, No. 4, July 1992)

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#### Materials strength testing machines

New materials strength testing machines can exert test loads up to 3/4 tons while requiring little space on a bench. *Features:* The machines provide edge-toughness testing, tension-, compression-, three- and four-point bend-testing facilities. They are suitable for materials research laboratories in industry, academic institutions and government organizations and for use in quality control applications. The ET500 is designed primarily for edge-toughness testing. Hard metals and ceramics can be tested for chipping resistance by making an indentation near the edge of the material and measuring the load required to cause the edge to flake. The CT5 is a vertical-loading compression- and tension-testing unit. The C50 is specifically for tablet hardness testing. (Engineering Systems, 1 Loach Court, Radford



Bridge Road, Nottingham, England NG8 1NB;  
Tel: 44 602 288708; Fax: 44 602 288715) (Source:  
*International New Product Newsletter*, October 1992)

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#### Thermal inspection of materials using artificial neural network system

A professor of the Mechanical Engineering Dept., Osaka Institute of Technology, and the National Aeronautics and Space Administration (NASA) of the USA have jointly developed an experimental system for inspecting defects of materials from the surface thermography.

An infrared beam is directed on the material, and the photographed temperature distribution analysed with an artificial neural network. Compared with the flaw detection method using ultrasonic waves, a conventional non-destructive inspection method, the new system enables measurements to be performed much more simply and rapidly. This new system can be applied to the non-destructive inspection of aerospace materials.

With the durabilities of large aircraft becoming longer than ever, the need for conducting non-destructive inspection of various body materials has increased. The exfoliation of bonded body parts is a cause of aircraft accidents, so the development of technology for the rapid inspection of bonded parts has become urgent. The non-destructive inspection system based on an infrared beam remote sensing technique is a possible solution.

With the thermal tomographic inspection method, a laser beam or an infrared beam from a lamp is irradiated on the surface of the target inspection material, and the temperature distribution on the material surface is photographed with an infrared ray camera for analysis.

If some flaw exists, the flow of heat from the material surface will be prevented from flowing further, and will appear as a fluctuation in the surface temperature distribution pattern. The purpose of this research project is to establish a system capable of learning the relationship between the temperature distribution fluctuation and the defects using a neural network.

A back-propagation (BP) method relating to the neural network's learning process was applied, consisting of a three-layered structure with a hidden layer between the input layer and output layer. In the experiments, a beam from an infrared lamp was directed against the material surface for a fixed time, and temperature images were collected as data showing temperature dispersion with time and position.

Two pattern vectors for the input layer were studied. One was the case in which the temperature

distribution with position direction was the input pattern, and the other the temperature changes with time in one position. The presence of defects was determined with the output layer. When two strips of aluminium plates with thicknesses of about 1 cm were bonded with epoxy resin and tested as the specimens, it was possible to detect defects at an accuracy of over 90 per cent.

Detecting the existence of defects in materials and determining the domains of defects are possible with relative accuracy by the learning effect based on the use of a neural network system, but good results are not necessarily obtained when using actual data. Therefore, using a hybrid inspection method may be effective, in which a general evaluation is made with the neural network system and, if necessary, detailed studies by the mathematical scientific method. (Osaka Institute of Technology, Mechanical Engineering Dept., 5-16-1 Omiya, Asahi-ku, Osaka 535, Japan. Tel.: +81-6-952-3131; Fax: +81-6-957-2134) (Source: *JETRO*, November 1992)

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#### Strength measuring system for composite materials

Shimadzu Corp. has started marketing a testing system called SAX-10 that applies tension and bending on composite materials such as glass fibre-reinforced plastics (GFRP) and, at the same time, enables the internal state to be observed on a TV screen by X-ray scanning.

Conventionally, the internal state of materials including internal destruction was estimated by measuring and estimating the sizes of cracks on the surfaces of materials. The rapidly increasing use of composite materials for sports and leisure goods such as tennis rackets and skis as well as aircraft rudders, steering wings and outer parts has made it necessary to observe and analyse in greater detail the state inside composite materials when large loads are impressed as well as the processes leading to the destruction of these materials.

SAX-10 is essentially a combination of a material strength tester and an X-ray TV system for observing the internal state of materials using X-rays. High-magnification, high-resolution imaging is possible of the materials during accurate strength tests, so the state of bonding and destruction of composite materials can be analysed most accurately. X-ray analysis at a magnification of 3-250 times is possible in real time and, by image processing, the images can be expanded to 500-1,000 times, so grains as fine as 15  $\mu\text{m}$  can be observed.

The testpiece can be observed in 360° so X-ray analysis is possible from any angle. The maximum load is 1 t, and loads can be impressed on testpieces at 0.1-1,000 mm/min during observations.

SAX-10 is applicable to composite materials and fabrics, artificial leather, rubber products including reinforced tires, the soldering of electronic parts which cannot be checked visually from outside, and assembled products with various parts. (Shimadzu Corporation, 1, Nishinokyo-Kuwabara-cho, Nakagyo-ku, Kyoto 604, Japan. Tel.: +81-75-823-1111; Fax: +81-75-811-3188) (Source: *JETRO*, September 1991)

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### Characterization of damage modes in impacted thermoset and thermoplastic composites

#### Introduction

Composite materials made of continuous carbon fibres and high performance polymers are gaining increasing acceptance in aerospace structures due to potential weight savings and efficient design considerations. These materials are being considered for primary (load bearing) structural applications in commercial and military aircraft. An important design consideration is low velocity impact by foreign objects (e.g., bird hits, runway debris, tool drop, hail, etc.). As the first generation of epoxy-based composites was extremely susceptible to impact damage (with attendant mechanical property losses), newer damage tolerant and damage resistant resins have been synthesized for composite applications. Laminated composites are known to undergo severe internal damage resulting from impact events that may or may not be evident from a surface inspection. Further, surface damage often offers an inadequate description of the complete damage state that exists within the laminate. Typical damage zones in impacted laminates consist of transverse matrix cracks,

delaminations, fibre failures and combinations of these. Impact damage in composites may result in a severe loss in load-bearing capacity, particularly with respect to post-impact compression.

In a previous study, a newly developed impact fixture was used to establish impact and compression after impact data on several composite systems. The goal of that study was to evaluate the impact damage resistance and residual compressive strength of various composite systems and to determine the effect of material characteristics on impact damage tolerance. Several composite systems reflecting generic categories of resin behaviour (such as brittle thermosets, toughened thermosets and amorphous and semicrystalline thermoplastics) were selected for this study. These materials possess widely different chemistries, cure/consolidation mechanisms, morphologies/microstructures and deformational capabilities. The present study focuses on mapping the resulting impact and post-impact compressive damage patterns in samples used in that study. The mapping was done with a view towards reconstructing the characteristic damage due to the impact event and eliciting key details of the damage mechanisms. Conventional fractographic techniques (optical microscopy and SEM) have been supplemented with novel ultrasonic techniques to achieve this characterization. This information, in conjunction with the mechanical data provides comprehensive information on the response of composite laminates to impact. (Excerpt from the *Journal of Reinforced Plastics and Composites*, Vol. 11, October 1992; article written by K. Srinivasan, W. C. Jackson, B. T. Smith and J. A. Hinkley)

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### 3. COMPUTERIZATION OF TESTING PROCEDURES

#### PCs help optimize materials testing

By computerizing their testing processes, manufacturers can "improve the quality of quality control", while saving both time and money.

Merging the personal computer (PC) with materials-testing equipment in the plant environment creates a cost-effective tool that is helping manufacturers compete in today's quality-conscious, international market. This article explores the capabilities, benefits and problems associated with computerizing materials testing, as well as the demands that are making automation of testing operations an economic and technical necessity.

#### Computer revolutionizes industry

Tremendous changes in both testing devices and standards have occurred during the 160-year history of materials testing in the United States. Only during the past few decades, however, have there been any significant improvements in the acquisition, analysis and archiving of test data. These gains are largely the result of rapid advances in the capabilities and versatility of computers.

The introduction in the mid-1960s of smaller, faster, easier-to-program third-generation computers marked the onset of a period of rapid growth in plant-wide automation. Within 15 years, the large number of new computer-hardware/software products and vendors made interconnection and communication standards a must. This demand for compatibility of information transfer among processes, test equipment and computers was met by the Manufacturing Automation Protocol (MAP) standard and the Open Systems Interconnection (OSI), developed under the unifying influence of the International Organization for Standardization (ISO).

By the 1980s, computer technology and supporting communications standards had advanced enough to provide a foundation for fully networked manufacturing systems. A major drawback, however, particularly for small and medium-size companies was the difficulty of applying the proper amount of "intelligence", or computer power, in a cost-effective manner.

The first computerized plants relied principally on mainframe computers, minicomputers and independent workstations, all of which carried high prices commensurate with their capabilities. Embedded microcomputers and programmable logic controllers (PLCs) managed most of the so-called intelligent process machines, but these control devices had (and still have) inherent programming and/or interface limitations.

Enter the personal computer. In the course of a single decade, the PC evolved from a backroom, spare-time project for a relatively few enthusiasts into a workhorse of the office, laboratory, and, finally, factory. Although its cost/performance ratio naturally positioned the PC in the gap between the PLC and workstation, much of the credit for its success in the lab and on the plant floor belongs to those backroom visionaries, who developed thousands of practical science and engineering programs.

#### PCs changed the face of testing

In most instances, those early individual efforts were driven by the need for a program or interface board that would solve a specific laboratory or measurement problem. As a result, many of today's materials-testing challenges can be met by off-the-shelf or turnkey systems. However, demand also is rising for better, more efficient methods of handling substantial increases in testing volume; coping with continued growth in the complexity of data-handling, data-analysis and test procedures; and providing optimized test procedures. Low cost, relatively high power, ease of use and reliability make the personal computer an ideal candidate for satisfying many of these requirements.

**Test volume:** A larger testing volume usually is the result of an increase in manufacturing productivity, a tightening of internal quality/specification controls to help ensure that products meet or exceed customer expectations and/or the need to characterize or verify the reliability of a new material/process. Today, significant increases in test volume also can be forced by new safety legislation, revised standards or more stringent product-liability insurance requirements. For example, the requirements of safety-related legislation that affect original-equipment manufacturers (OEMs), such as aircraft, automobile, and heavy-equipment companies, often become part of the OEM's test specifications for raw materials or purchased parts. This shifts the burden for proving quality from the user's incoming-inspection department to the vendor's quality-assurance staff. Consequently, many heat-treating shops, fastener plants, foundries, steel mills and other material/component suppliers are having to implement new testing and statistical process/quality control (SPC/SQC) procedures. Product value and quality increase, but so too do the number and complexity of tests and the amount of test documentation required.

These expanded testing needs can be accommodated via computerization, often without having to add people. Proper use of computer resources can even lead to a reduction in the labour content of testing processes.

**Data availability:** The true value of test data lies in their ability to isolate out-of-tolerance product, so it can be immediately removed from the manufacturing system. The earlier in the production process that a non-conformance is located, the less time and money are lost due to scrap and rework. When used in combination with efficient computer-to-process communications, the rapid access to test data provided by computerized testing can assist in quickly bringing a wayward process back under control. This is especially valuable in large facilities, and critical in those using continuous process systems.

In addition to its swift-response capability, computerized data logging makes it possible to efficiently record and maintain the large amount of data needed for the historical analyses that provide the basis for SPC/SQC. With the addition of networked computer communications, that information can be made immediately available to a mainframe, other PCs, or terminals, which, in turn, can be used by managers, engineers and manufacturing and marketing personnel to obtain detailed information about the quality of a particular part or product.

Even at the single-PC level, it is reasonably easy to produce high-quality reports and statistical graphs for internal use or to submit to the customer. Data also can be transferred to other locations within the plant or around the world via diskette, modem or fax.

**Test optimization:** The application of the PC to mechanical, dimensional, metallographic and spectrographic test/analysis has resulted in significant gains in productivity. Actual operator interface time (hands-on time) can be dramatically reduced for tests that involve a large amount of repetitive action or have lengthy hold times between process steps. Many mechanical-testing applications meet one or both of these criteria. Jominy end-quench hardenability testing and case-depth analysis, for example, benefit substantially from computerization. Done manually, a conventional single-bar, single-sided Jominy analysis takes approximately 20 minutes to perform, including data recording and graphing. An automated, three-bar Jominy system does all this and more - for all three bars - with an operator interface time of less than 10 minutes. The computerized system runs the tests, analyses the data, performs "A to Z" comparisons on both sides of three Jominy bars, prints and archives the data, graphs the results (H-bands and hardenability indices included) and summons the operator if necessary.

Case-depth analyses offer similar opportunities for time savings, especially when microindentation hardness-test methods are used. An automated, 20-data-point Vickers case-depth analysis has an operator interface time approximately 12 minutes less than that needed to manually run a similar test.

In both of these examples, efficiency is the key word. While the machine is performing the test, the operator is free to work at other tasks. The computer also relieves the operator of the time-consuming tasks of data logging and archiving. Reports and graphs can be automatically generated, and data are stored for easy retrieval, transfer to a mainframe or incorporation into a database, such as an SPC/SQC system or laboratory information management system (LIMS).

#### PCs boost testing capabilities

Sophisticated data-analysis systems such as these are needed to satisfy the increasingly complex testing requirements of many manufacturers. For example, a major supplier of aircraft engines needed a very narrow-band case-hardness test for a part having a total hardened depth of about 2.5 mm (0.1 in.). The case-depth analysis technique specified is the 15° taper-grind procedure (per SAE J423a). This normally straightforward analysis was complicated by two additional requirements: the case depth report and accompanying graph had to read in actual depth, and the case depth had to be calculated as a function of core value plus 120 Vickers hardness points. The challenge was readily met using an off-the-shelf programme having a built-in math package and macro language. No customization was required.

This ability to solve complex analytical problems with a few keystrokes also is provided by the computer packages offered with many metallographic, spectrographic, coordinate-measuring, image-analysis, and laser-interferometer systems, among others. Assuming proper setup and factory support, the capabilities of the "tool-box" available to materials-analysis laboratories generally are limited only by the requirements of the test and the imagination of the user.

**Repeatability:** In most instances, a computer "assistant" also will help to ensure repeatability. Proper structuring of the program will lead the operator through each step in the same order every time. No steps will be omitted, overlooked or compromised. If an operation can be placed under total computer control, the human-error factor also can be eliminated. However, the degree of computer control possible depends on the test. Hardness testing and chemical analysis often can be automated, even to the extent of incorporating robots. Metallography, on the other hand, usually requires human interpretation of results.

**Less training:** Another major advantage of automated testing relates to the computer's ability to store and apply a great deal of information about the procedure, which helps compress the operator's learning curve, reduce the entry-level knowledge required to run the system, and compensate for the variability among different operators. For example, the computer can be

relied upon to control test parameters, preselect machine behaviour patterns, perform mathematical calculations and assist the operator via user-designed prompts and help screens.

A well-designed program allows supervisors to construct and name discrete test sequences, with no limitation except disk storage space. By adopting naming conventions that clearly identify the part being tested, and by including prompts in the sequence, even inexperienced technicians are able to initiate and run any test using a minimum number of keystrokes. This means that a single operator can handle a variety of tests and test instruments, and that occasional users can run tests with little or no supervision. Detailed knowledge of test procedures is not required. That information is distributed between operator and PC and often is enhanced by the computerization of engineering-level expertise (expert systems).

Although adding a PC to a test set-up costs from \$2,000 to \$6,000, the investment can be expected to be repaid many times over. If well planned, computerization can reduce recurring costs associated with labour and error. More to the point, if a PC can help prevent costly scrap or rework, or if it can enhance product quality, and thus the company's competitiveness, no further justification is required.

#### Basic PC selection factors

The price of a PC is very reasonable considering the return on investment. Prices vary over a wide range, however, and the most cost-effective PC is not necessarily the one with the highest feature-per-dollar ratio. Complete compatibility with certain well-established standards, for example, can be worth several times the "savings" gained by purchasing a non-compatible unit.

The most important factor to be considered when selecting a PC is how it will be used. Today, the basic PC for general-purpose laboratory applications has an 80386SX microprocessor (Intel Corp., Santa Clara, California), 2 to 4 megabytes of RAM (random-access memory), a 40 to 80 megabyte hard-disk drive, and a high-resolution, video-graphics monitor. This combination provides the memory, speed and graphics capability required by many analytical programs, as well as a reasonable amount of data-storage space on the hard drive. PCs intended for specific tasks, or embedded in a test system, often are based on the less powerful Intel 80286 or 8088 chips. They also cost less. Other choices that have to be made concern the bus standard, BIOS (basic input/output system), resistance to the industrial environment (degree of "hardening") and manufacturer.

PC bus standards govern the physical layout of the circuitry and accessory plug-ins. The market currently is dominated by two IBM (International Business

Machines Corp., Armonk, NY, USA) systems: ISA (Industry Standard Architecture), used for the IBM PC, XT, AT, PS/2 models 30 and 40, and compatibles; and MCA (Micro Channel Architecture), used for the PS/2 model 50 and higher. There are considerably more board-level products available for the ISA bus, and users seem to prefer it over the MCA system. Other architectures include EISA (Enhanced Industry Standard Architecture), the Apple/Macintosh bus (Apple Computer Inc., Cupertino, California), and various workstation and microcomputer buses that fall outside the range of this discussion. Like the MCA bus, the Apple/Macintosh architecture does not yet have the same high level of third-party hardware support that the ISA bus enjoys. However, Macintosh's excellent user interface and recently expanded plug-in capability give it the potential to be a major contender in the testing market.

The principal purpose of the BIOS is to serve as the interface between higher-level applications software and PC components. While several systems provide full compatibility with all software designed around the IBM PC format, there also are many that have been altered to provide special features. These modified systems can cause problems when used with software that requires 100 per cent IBM compatibility. If you are considering purchasing a computer that does not provide *total* compliance with an accepted bus or BIOS standard, check with potential software or board suppliers to ensure compatibility in the intended application.

The physical environment in which the computer must operate is not necessarily a limiting factor. Most applications can be handled by "off-the-shelf" computers, and there are several companies that specialize in "ruggedized" or hardened PCs that can, for example, be hosed down with water or operate beside a blast furnace. (Standard desktop units normally are not recommended for use on the factory floor.)

#### Proper preparation precedes purchase

Although automating any test method theoretically can provide a positive cost/benefit result, success is not guaranteed. Several precautions should be taken to avoid the pitfalls commonly encountered when purchasing a computerized system.

Fundamental to a successful automation venture is gaining a thorough understanding of the exact requirements of the particular testing/information-handling application and how it relates to the overall manufacturing process. The analysis should include current and future needs, projected over the system lifetime or its estimated payback period, as well as anticipated levels of system performance, utilization and flexibility. (It often is helpful to consider any computer purchase as if the new unit was going to be a node on a network consisting of all the computers in the facility.) Vendors need this information to configure a system that

will meet your minimum requirements. The analysis also may help to shorten the list of potential vendors.

Hands-on demonstrations of the software or peripheral hardware being considered will provide a solid basis for comparison. For applications software, look for a well-written program having a user-friendly interface and a well-organized, easy-to-read and unambiguous instruction manual. The best programs tend to be those whose use is intuitively obvious. This "try before you buy" approach is particularly essential in light of the proliferation of vendors and packages available for any given task. For example, the number of vendors of quality-related software in one trade magazine's buyer's guide climbed from 78 in 1983 to 165 in 1991.

A third recommendation is to contact current users of the hardware and software products being considered for purchase. Software, in particular, is complex and hard to debug. The best indication of high quality is long-term, relatively trouble-free use by others in applications similar to your own.

#### **"Factory of the future"**

About 15 years ago the "factory of the future" concept first made headlines. Today, with robots working on the production line and in the laboratory, and with computers helping to monitor and control the operation of entire factories, we are approaching that ideal.

Materials testing is one method of closing a process-control loop and minimizing, if not eliminating, the production of nonconforming products and their associated costs. The use of automated testing systems that combine cost-effective PCs with intelligent test equipment significantly increases our ability to keep processes under control and to spot out-of-control processes before too much damage has been caused. Computerization also provides faster, better documented and more repeatable tests, which are much less dependent on the skill of the operator. Properly applied, computerization is a powerful tool for improving the quality of quality control.

For more information: Wilson Instruments, 6 Emma St., Binghamton, NY 13905; Tel.: 607/770-4500; Fax: 607/770-0109.

#### Components of a basic computer system

Buying a computer is a difficult task. For example, a selection table in a recent buyer's guide listed

the features of more than 100 different PCs. Major system components and options are briefly described here.

#### **Processor\***

- **8088** (640-kbyte memory/8-MHz speed): Used for low-level word processing, statistical process control (SPC), and business applications; simple machine control. Speed, memory addressing and I/O (input/output) addressing capabilities are limited.
- **80286** (4 Mbyte/10 MHz): Used for complex word processing, data acquisition, machine control, statistics and curve fitting programs; simple image-analysis applications; and small databases.
- **80386SX** (16 Mbyte/20 MHz): Used for Microsoft Windows (Microsoft Corp., Redmond, Washington) applications, basic CAD (computer-aided design), math-intensive and complex graphical analyses, image-analysis applications and medium-size databases.

\* Designations are those of Intel Corp., Santa Clara, California.

#### **Coprocessor**

Provides a significant speed advantage in applications that have complex math calculations or graphics.

#### **Memory**

Programs that have to calculate many variables or control many devices usually require a large memory. The amount of memory that is directly accessible depends on the processor. A rule of thumb: running more than one task under Windows (or another shell) requires from 500 kbyte to 1 Mbyte per application, plus that required by the shell.

#### **Hard disk drive**

Like memory, the required disk-drive capacity also depends on the applications. For example, an 80- or 120-Mbyte drive may be needed to handle the requirements of a laboratory information management system (LIMS). Drives tend to fill up fast, so it is better to have excess storage capacity. For example, a typical 40-Mbyte hard drive used for materials-testing applications contains these files:

Program	Allocation Mbyte
DOS (disk-operating system)	2
Windows	3
Spreadsheet	3
Word processing	4
Data acquisition	1
Machine control	1
C compiler with assembler	15
Text editor	0.5
Miscellaneous utilities	4
Available for data files	6.5

**Tape backup**

Strongly recommended to provide backup in case of disk failure. Should match disk size.

**Graphics adapter**

- CGA: Recommended for text applications, simple graphics programs;
- VGA: Recommended for Windows applications, graphics, icon-based programs, basic CAD;
- SVGA or XGA: Recommended for CAD, visualization, and virtual-reality applications.

(Source: *Advanced Materials & Processes*, November 1991)

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Computerization of data is making an impact on non-destructive testing (NDT) standards and instrumentation

Clearly, advances in computer equipment and techniques have had a strong impact on non-destructive testing (NDT) and the standards for this important quality control technology. ASTM Committee E-7 on Non-destructive Testing has been addressing the issue of computerization of data for NDT and several new standards are well advanced. This review summarizes recent work and indicates several directions that NDT standards will take in the immediate future. A new,

extended review of NDT standards is now available for additional information.

The computer era has had a significant impact on NDT equipment, permitting manufacturers to design and offer smaller, easier-to-use equipment with improved features. Indeed, some new NDT instruments are made by adding an extra board to a personal computer. Although this new breed of NDT equipment frequently produces results that are directly comparable to the predecessor analog versions, the use of digitized data formats and advanced imaging technologies has brought about the need for computer-related standards for image reconstruction, digitization of data, and storage, retrieval and exchange of data. These important issues are currently being addressed by Committee E-7.

The need for digital image reconstruction and the computer-based handling of large volumes of data is exemplified by computer tomography. This advanced diagnostic procedure, originally developed for medical applications, has now entered the industrial inspection market. Standards work has been under way only a short time, but a new Standard Guide for Computer Tomography (CT) Imaging is now available as ASTM Standard E 1441. This document provides general guidance for potential users of CT imaging for NDT. A new document, Standard Practice for Computed Tomographic Examination, now in ballot in Committee E-7, provides application details for computer tomographic examination. Obviously, the use of computer tomography requires the handling of an extensive base of digital data.

The intricacies of digital data handling and manipulations utilized by this technique has introduced a whole new lexicon of unique terms and definitions. Many of these new terms are also being balloted in Committee E-7 in an effort to provide the basis for communication of some of the concepts associated with digital image handling.

The new capability to digitize NDT data has been recognized in terms of needs for storage media for NDT data. A new Standard Guide for the Storage of Media that Contains Analog or Digital Radioscopic Data (E 1453) has recently been issued. The guide provides a summary of recommended methods to store databases on magnetic and optical media. This topic was recognized as important and ASTM issued standards on real-time, electronic X-ray examination and radioscopy. The amounts of data that can be collected with this fast-response examination method prompted members of E-7 to prepare this new standard. Similar issues are recognized with the new capability to digitize the data on a radiographic film. A 14- by 17-inch (356- by 432-mm) film digitized at the 12-bit level (4,096 grey levels) at a spatial resolution of 50 micrometres yields an image data matrix of 17,200 by 14,100, or 364 megabytes. This capability is now commercially

available and has been recognized by the American Society of Mechanical Engineers.

The large databases, as indicated above, lead to many standards issues. The data must be stored on an accepted media, made available for retrieval and display, and must be confirmed in some way as to the faithful representation of the original data. In addition, these large databases lead to questions concerning data compression and the capability to exchange or transfer data. All these issues for computerized NDT data are under discussion in Committee E-7.

Among the E-7 subcommittees considering computerized data is E07.02 on Reference Radiological Images. This subcommittee has issued 13 standards that provide reference radiographic images of welds and castings. These documents contain over 200 pages of reference radiographs consisting of almost 1,200 radiographic images, illustrating grades and types of defects. The reference radiograph documents are widely used standards. Although there is a continuing strong demand for these film documents, the members of E07.02 recognize that there will be an increasing need for electronic, digital representations of radiological data from digitized film, radioscopy and tomography. These new, important topics are under consideration.

Recent progress can be reported on standards for the transfer of digital data. ASTM Committee E-7, working closely with Committee E-49 on Computerization of Material Property Data, has prepared two new standards describing the information needed to transfer digital test data from ultrasonic and radiological images between machines using dissimilar data formats and structures. The new Standard E 1454, Guide for Data Fields for Computerized Transfer of Digital Ultrasonic Testing Data, outlines 124 data fields to describe the data from a digital ultrasonic test. The data fields needed include descriptions of the examination system, details of the ultrasonic equipment and the examined part, and parameters of the data. Additional information about the topics of the data fields for digital ultrasonic testing is given in table 1. Although all 124 data fields are desirable to permit a full understanding of the transferred ultrasonic data, 40 data fields are deemed essential to understand the transferred data. A similar standard is being readied for ASTM issue, Guide for Data Fields for Computerized Transfer of Digital Radiological Test Data (E 1475). This guide describes 93 data fields (45 essential data fields) to accomplish the transfer of digital radiological images obtained from radiographic or radioscopy examinations.

This indicates progress in the transfer of digital ultrasonic and radiologic data. Similar procedures can be used to transfer digital data from other NDT systems, such as eddy current, infrared and others.

The data transfer among dissimilar systems followed E 1454 and was compatible with a proposed new international standard, Standard for the Exchange of Product Model Data (STEP). This represents a starting point for bringing the transfer of NDT data into the extensive international work involving the transfer of data for product manufacturing and life cycle. This new international data transfer work (STEP), building on work in the United States on the Product Data Exchange Specification (PDES, now Product Data Exchange using STEP), is envisioned as an important part of the future for work in product manufacturing and life cycle. The data exchange specification will permit incompatible systems to communicate through a neutral, intermediate data exchange, as illustrated in figure 1 (page 53). This approach provides protection of proprietary data handling information and easy entry into the system in that only two translators need to be prepared for each new unit.

This brief review confirms that computerized data is now an important part of NDT standards and that it will be come increasingly important in the future.

Table 1

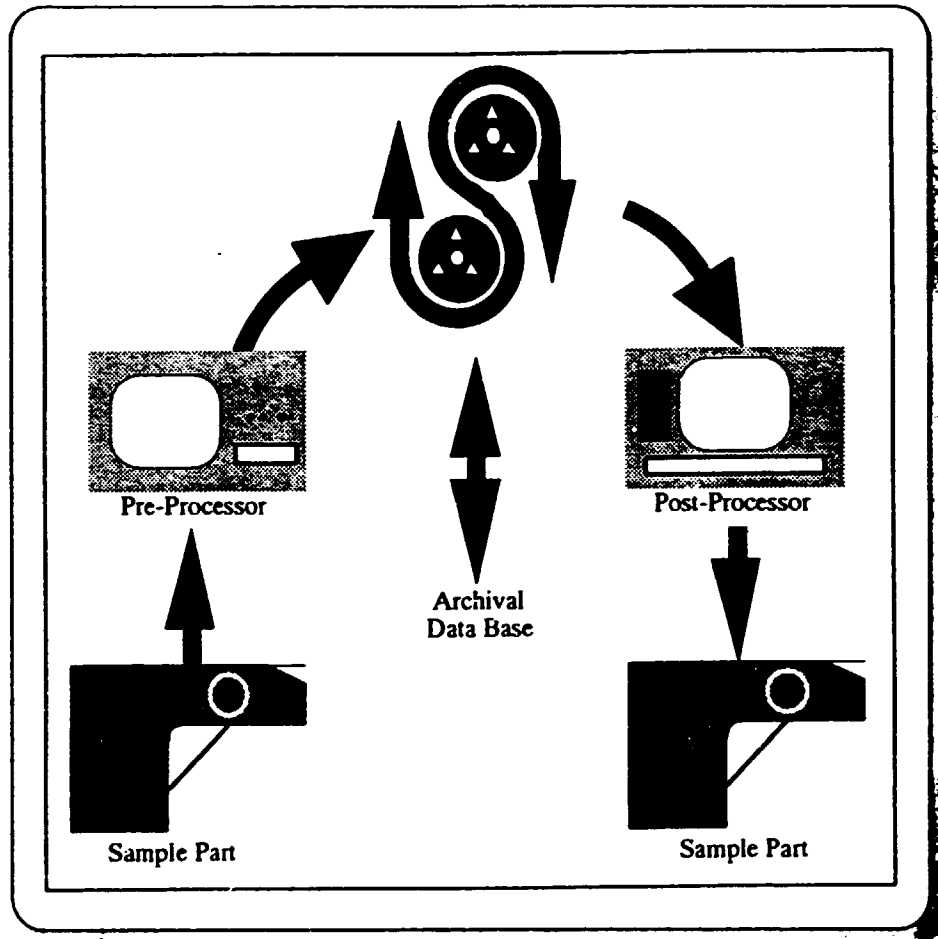
**Broad subject headings for digital ultrasonic data fields**

Header Information
Examination System
Pulser
Receiver
Gate
Search Unit
Examined Sample
Coordinate System and Scan
Examination Parameters
Examination Results



Figure 1

Illustration of the Intermediate Exchange Approach



(Source: *ASTM Standardization News*, July 1992)

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#### 4. STANDARDIZATION OF TESTING AND EVALUATION

##### Standardized testing of advanced composites

Major advancements in composites technology have evolved during the past 10 years, including new developments in polymer resin formulations, fibre reinforcements, and processing, which have led to increasing use of advanced composites, especially in aerospace applications. The increasing interest of designers in these materials is based on the advantages of composites over other materials including higher strength-to-weight ratios (specific strengths), higher modulus-to-weight ratios (specific moduli) and better fatigue resistance.

Despite these attractive properties, one obstacle to greater use of advanced composites is the lack of standardization - the adoption and use (by consensus or decision) of engineering criteria applied through standards and specifications. Specifications and standards are the main elements of a standardization process. In the composites industry, there are no universally standardized mechanical, chemical, or physical test methods.

Specifications and standards for plastics and advanced composites are used by industry, government, and universities in research and development, design, manufacturing, procurement, and maintenance. Standards are essential to generate design data and for standards reliability. They also are needed for quality control, product specification or definition, and materials testing; quality-control standards are essential in manufacturing processes to ensure high product quality and low rejection rates. In addition, standard test methods also assist in the ability to report materials properties consistently and to qualify new materials for use in various applications.

Depending on the extent of testing and intended use, the cost of qualifying a new composite material for a specific programme could range from \$50,000 to \$10 million. Various government agencies and industry organizations recognize and are addressing the problem of high qualification costs and the need for standardized composites testing to develop uniform specifications and material-property databases.

In the 1980s, a new industry association, the Suppliers of Advanced Composites Materials Association (SACMA), was formed and, in 1986, established a task force composed of representatives from materials suppliers, users, and testing communities to develop a cost-effective, specific, and statistically significant set of methods and procedures for testing composite materials. SACMA is not a standards-writing organization, and as a trade organization, does not have the charter or

resources to develop and maintain consensus standards. Rather than developing standards, or creating design allowables or manuals, SACMA goals are to:

- Raise the level of understanding throughout the advanced composites industry of the importance of specifics in test methods;
- Reduce materials characterization and qualification costs;
- Develop a set of cost-effective, specific, credible, and statistically significant test methods and procedures, where such test methods and procedures do not already exist;
- Facilitate industry use on certain key test methods and procedures.

##### Standardization barriers

According to SACMA, testing needs of the advanced composites industry generally are driven by user (aerospace companies) requirements, i.e., each company develops its own set of test methods to characterize and qualify a material, and to establish design allowables. Thus, while the need to know basic materials properties is essentially the same for all users, methods used to determine these properties are different and do not always yield comparable results. Compounding the problem is the lack of a thorough understanding of many test methods due to the large number of tests and test variations from various US standards-writing organizations (ASTM, Department of Defense, and National Aeronautics and Space Administration, for example), universities, and materials suppliers, as well as those from organizations in Europe and Japan.

As an example, a comparison of 30 prepreg specifications shows the use of 17 different compression-test configurations. Besides complicating compliance with customer material requirements, multiple test methods create problems and hidden costs including:

- Cost to purchase and maintain many different test fixtures;
- Voluminous paperwork required to ensure use of the appropriate method;
- Extra training to develop expertise for each method;
- Difficulty or inability in combining test results into a common database.

### The SACMA approach

SACMA established the Test Methods Task Force to address the common problems that exist in standard test methods; that is, very general test descriptions that can apply to a broad variety of materials and laboratory procedures; and often-missing details needed to successfully and reliably perform the test on specific materials. To handle the latter problem, the task force focused on conventional carbon fibre-reinforced prepreg materials because this was the area regarded to have the greatest need for standards and also because task-force members had the greatest expertise with these materials.

Nine SACMA recommended methods (SRMs) for testing composite materials were released in 1989 for industry review. They are:

- SRM 1-88 Compressive Properties of Oriented Fibre-Resin Composites;
- SRM 2-88 Compressive After Impact Properties of Oriented Fibre-Resin Composites;
- SRM 3-88 Open-Hole Compression Properties of Oriented Fibre-Resin Composites;
- SRM 4-88 Tensile Properties of Oriented Fibre-Resin Composites;
- SRM 5-88 Open-Hole Tensile Properties of Oriented Fibre-Resin Composites;
- SRM 7-88 In-Plane Shear Stress-Strain Properties of Oriented Fibre-Resin Composites;
- SRM 8-88 Apparent Interlaminar Shear Strength of Oriented Fibre-Resin Composites by the Short Beam Method;
- SRM 10-88 Calculation of Fibre Volume of Composite Test Laminates;
- SRM 11-88 Conditioning of Composite Test Laminates.

SRMs 1, 2, 3, and 5 have been submitted to ASTM for standardization. No generally acceptable method has been found for SRM 6-89 (Flexural Properties of Oriented Fibre-Resin Composites), while SRM 9-89 (Bearing Properties of Oriented Fibre-Resin Composites) is still under development.

The recommended methods were developed to identify critical parameters in the testing of very complex materials, and because of the extremely high strength and moduli of these materials, testing and test-specimen quality are closely controlled. To facilitate

SRM implementation, each method incorporates a common format (patterned after ASTM standards) to maximize comprehension, clarity, and use, and to minimize changes when standardized. SACMA Recommended Methods are not "national consensus standards". However, they represent an association consensus, and SACMA hopes to obtain ASTM approval of the new methods, as well as ASTM modification of existing standards that need to be defined better.

The recommended methods are the result of a coordinated effort by over 14 composite material suppliers and parts manufacturers who reviewed all available composite airframe-material specifications, selected the most appropriate specification elements, and compiled them into a single document. Where it was possible to use an existing standard as a basis, the task force attempted, based on experience, to conform to the requirements of the existing standard, making changes leading to increased specificity. In addition, regarding test variables, the task force chose the option that was the simplest, least ambiguous, and the one that should result in the best precision.

For example, a common element adopted in the SRMs is the definition of modulus as the secant modulus determined between two distinct points on a load-strain curve. This definition was recommended because advanced composites are not linear in load-strain behaviour, and recording specific loads at specific strains is much more precise than drawing lines at arbitrarily selected points to determine the slope. By comparison, typical standards, such as those written by ASTM, do not define the details of modulus determination, which leaves the choice of modulus (tangent or secant) and its location on a non-linear stress-strain curve to the discretion of the individual conducting the test. While this situation allows testing flexibility, it makes comparison of data from different sources difficult or impossible.

The development of these nine initial SRMs is intended to be a starting point to foster further standardization through the efforts of SACMA and other industry and government organizations.

### Materials standardization

According to SACMA, the rapidly increasing use of carbon fibres in structural and heat-resistant composite applications in the aircraft/aerospace and defence markets has increased the need for materials standardization. However, carbon fibres will not become standard or commodity products in the near future due to the complex, proprietary nature of their manufacturing processes. If carbon-fibre manufacturing processes and resulting products cannot be standardized, it becomes necessary for materials suppliers to standardize product descriptions through the use of common lot-acceptance procedures and certification-test methods. This situation presents problems similar to

those addressed in testing advanced composites; i.e., the existence of too many diverse, redundant specifications, standards, and acceptance-test methods, which leads to confusion and high costs for both users and suppliers in maintaining multiple test capabilities, and generates uncombinable data.

To help alleviate this situation, the SACMA Carbon Fibres Test Method Task Force, comprising quality control specialists from major domestic and international carbon-fibre manufacturers developed a generic lot-acceptance procedure (including a unified statistical sampling plan based on Mil-Std-414) and five recommended carbon-fibre test methods.

Methods released in 1990 are:

- SRM 12-90 Lot Acceptance of Carbon Fibres;
- SRM 13-90 Mass per Unit Length of Carbon Fibres;
- SRM 14-90 Sizing Content of Carbon Fibres;
- SRM 15-90 Density of Carbon Fibres;
- SRM 16-90 Tow Tensile Testing of Carbon Fibres;
- SRM 17-90 Twist in Carbon Fibres.

The SRMs describe the necessary apparatus, test conditions and tolerances, detailed procedures, retest criteria, data calculations and precision, and test-record requirements. Of special note is an attempt to standardize the impregnated-tow test, which has been of concern to the advanced composites industry because a common resin system and consistent modulus-measurement techniques are not used by all carbon-fibre manufacturers. SRM 16-90 takes this situation into consideration by specifying both an impregnating resin and cure cycle as well as defining secant modulus as the relationship of load to strain at two specific strain points on the stress-strain curve. It also requires that elongation between the points be determined using a direct strain-measurement device (extensometer). The recommended method is more precise than determining fibre elongation by test-machine cross-head travel, which must be corrected for system compliance.

Full conversion to the use of the carbon-fibre test methods is targeted for early this year; this follows round-robin testing, presentation to customers, and parallel testing and database generation. SACMA also is seeking ASTM and SAE approval of the SRMs, as well as revision of existing consensus standards applicable to acceptance and certification testing of carbon fibres (see diagram on page 59).

Specifications standards uses

**Research and development**

- Material characterization

**Design**

- Development of material-property databases (design allowables)
- Qualification testing of materials
- Materials selection

**Manufacturing**

- Processing
- Quality control
- Safety
- Reliability

**Procurement**

- Specification of requirements
- Quality-assurance acceptance/rejection
- Legal instrument in contracts

**Maintenance**

- Repair

For more information on SACMA SRMs: 1600 Wilson Blvd., Suite 1008, Arlington, VA 22209, USA, Tel: 703-841-1556, Fax: 703-841-1559. (Source: *Advanced Materials & Processes*, February 1991)

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The value of ASTM\* to the commercial fatigue testing laboratory and its customers

Standardizing test methods for the advancement of fatigue knowledge

The contract fatigue testing laboratory provides a valuable service to a variety of industries including those involved with aerospace, transportation, power generation, structures, prosthetics, orthopaedics and electronics. While many companies within these industries maintain fatigue testing capabilities of their own, they have found it economically beneficial to farm out an increasing volume of testing. Two basic viewpoints on subcontracting of fatigue testing have evolved. The predominant viewpoint is to have any test that becomes standard performed by an outside

\* ASTM: American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103, USA.

for the development of techniques critical to the testing of advanced materials and components. The opposing laboratory. This allows the company to utilize its talents; viewpoint is to keep standard testing in-house, providing for consistency and quick throughput, while relying on outside expertise for the development of non-traditional test techniques.

Because of the increased volume of fatigue testing in the 1960s and early 1970s, it became apparent that standardization of test methods was necessary. The guidance that ASTM Committee E-9 on Fatigue has provided in standardizing such methods has resulted in increased usage of the commercial fatigue laboratory, a decrease in testing costs and the generation of significant quantities of accurate, reproducible data.

### What is fatigue?

ASTM Standard E 1150 defines fatigue as "the process of localized permanent structural change occurring in a material subjected to conditions that produce fluctuating stresses and strains at some point or points and that may culminate in cracks or complete separation after a sufficient number of fluctuations". The most important consequence of fatigue damage is that it may occur at fluctuating peak stresses or strains well below those which would produce failure under static conditions.

Fatigue is sometimes divided into two somewhat loosely defined regimes. Low-cycle fatigue generally includes cycles to failure below 100,000, although this limit is sometimes defined as 10,000 cycles. Peak stresses exceed the yield strength and significant plastic strains may be generated. High-cycle fatigue results in cycles to failure exceeding 100,000, and the strains produced are usually all elastic.

### The fatigue test

Fatigue tests are typically characterized according to loading mode. Bending fatigue represents the most common of fatigue testing methods. Two types of bending tests are commonly used:

- *The rotating beam machine* employs an electric motor which rotates a cylindrical specimen, normally at a frequency of 60 hertz. A dead weight load is applied to the centre of the test specimen producing a moment that causes bending of the sample. A fully reversed sinusoidal loading is thereby obtained. (Maximum and minimum stresses are of equal magnitude and opposite sign.)
- *Plate bending cantilever bend machines* use an eccentric crank to cycle the specimen at a constant displacement. This permits the

application of a mean stress, an important consideration in fatigue life.

While bending fatigue testing is relatively simple and inexpensive, it does have significant drawbacks. Stresses and strains are not directly measured: the rotating beam tests require calculation of stresses via the moment; calculation of stresses for the plate bending tests are based on the assumption of completely elastic behaviour, an assumption that rarely holds true, even on a macroscopic level.

While bending fatigue tests subject only the outer fibres of the test sample to maximum stress, the axial fatigue test subjects the test sample to a uniform stress through its entire cross-section. Additionally, the axial test allows for the direct measurement of stress and strain. Because of these advantages, axial fatigue test results are often used for the generation of stress-life  $S-N$  and strain-life  $\epsilon-N$  data for fatigue life predictions.

### The axial fatigue testing machine

Closed loop servo-hydraulic and electro-magnetic resonant test machines are the systems of choice in most commercial fatigue laboratories. Closed loop systems employ a feedback loop that continuously compares the command and feedback signals. The servo-controller reacts to the difference between these two signals and applies a correction to the system. With high system gain, test parameters are continuously controlled.

Servo-hydraulic test systems offer the greatest versatility in programming and control. Test frequency and waveform can be adjusted, and force, displacement and strain can be controlled. Hydraulic rams can easily provide the required forces.

Resonant systems operate at relatively high frequencies and produce sinusoidal waveforms. This high frequency capability permits the completion of long-life tests in relatively short duration.

### Why perform fatigue tests?

In general, fatigue tests are performed for one of four reasons:

- *Quality control or quality assurance* - Testing may be performed on in-process materials or products or on a specified sampling of finished parts to ascertain conformance to specification requirements;
- *Research and development* - Fatigue behaviour of developing materials is evaluated to provide early design data;
- *Fitness for use* - Fatigue testing is performed to establish the suitability of a material or

components to meet the requirements of a newly defined use; and

- *Referee or round-robin* - Testing is performed to verify the accuracy of equipment and methods, and the competence of testing personnel.

#### Concerns specific to the contract laboratory

Because they service numerous customers, contract testing laboratories must address a number of particular concerns. All test data generated are the property of the customer. As such, the proprietorship of these data must be safeguarded.

Although most orders reference ASTM or other industry standards, many companies have unique testing requirements. A quality department, explicitly familiar with the laboratory and focused on the needs of the customers, ensured that procedures are developed and implemented to meet these requirements. Quality and technical audits by customers from various industries promote continuous improvement of quality systems and test techniques.

#### Qualifications of the contract fatigue testing laboratory

Generally accepted requirements exist for all testing laboratories. A comprehensive quality system must be documented via a quality manual as described in International Organization for Standardization (ISO) 9003. ASTM Standard E 548 establishes the criteria for evaluating laboratory competence. In addition to the aforementioned requirements, accreditation by specific companies or agencies may be required. Participation in industry-wide round-robin testing programmes can provide supplementary data demonstrating laboratory proficiency.

#### ASTM standards and activities

ASTM standards are consensus standards. The fatigue testing standards adopted by ASTM Committee E-9 have been developed through the cooperative efforts of the researchers who utilize the fatigue data, the equipment manufacturers, and the testing laboratories that generate the data. The result of this unusual marriage of capabilities is the evolution of utilitarian standards documents that are realistic and useful. This "concurrent engineering" philosophy has

been employed by ASTM for years. "Corporate America" has more recently adopted this concept as part of the "new" ideology known as "Total Quality Management."

Current standards for fatigue testing under the jurisdiction of Committee E-9 (E-9 recently joined with E-24 on Fracture Testing to form Committee E-8 on Fatigue and Fracture) include ASTM E 466 on load-controlled fatigue testing, E 467 on verification of dynamic loads, E 468 involving presentation of fatigue results, E 606 on strain-controlled testing, E 739 on the analysis of fatigue data, and E 1150 containing definitions relating to fatigue.

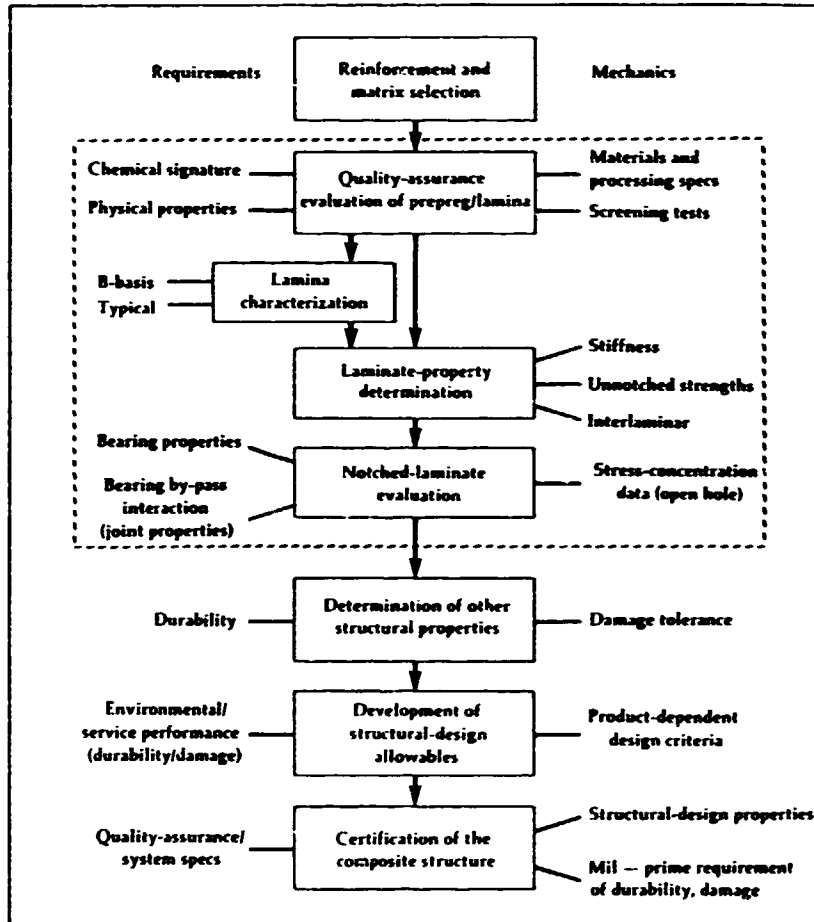
ASTM activities afford the commercial fatigue laboratory the opportunity to compare its techniques with those of others through ASTM-sponsored round-robin testing programmes. Twenty laboratories participated in a recent strain-controlled, low-cycle fatigue round-robin involving the testing of American Iron and Steel Institute 316 stainless steel. This programme was initiated to evaluate the application of ASTM Recommended Practice E 606. Another round-robin programme currently being conducted involves thermal mechanical fatigue testing, a special application of low cycle fatigue in which the specimen temperature is varied during the loading cycle. This complex test requires the ability to separate the temperature-induced strain from the mechanical strain. Preliminary results show remarkable agreement among participants. Currently being planned is a round-robin programme to apply strain-controlled test techniques to a metal matrix composite material.

ASTM standards also have great importance to the customers of the commercial laboratory. Data generated by adherence to such standards have significant advantages. Comparisons to previous test results or to data published by other companies become meaningful. The necessity of performing redundant testing is eliminated.

Involvement in ASTM activities is both an obligation and a privilege. ASTM creates a forum for the exchange of ideas. This exchange is necessary to the continued advancement in fatigue knowledge required to support new materials and designs (see diagram on page 59). (Source: *ASTM Standardization News*, March 1993)

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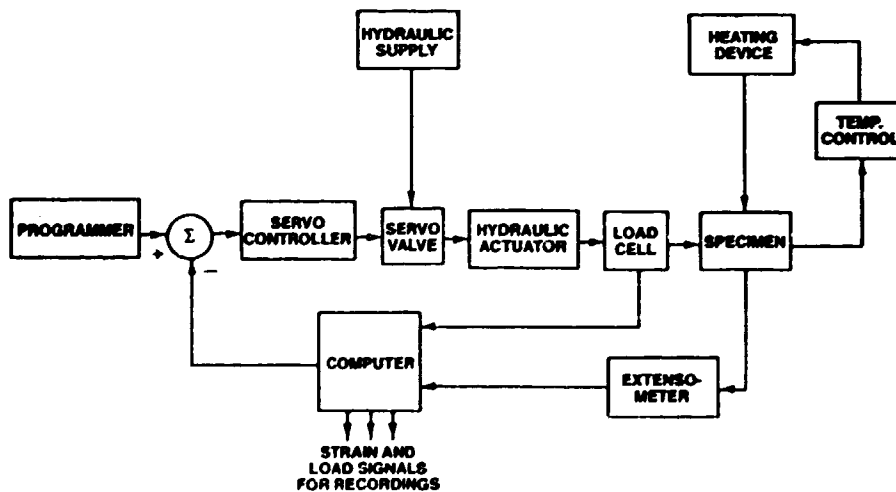
Qualification of composite materials and structures for major defence systems, such as aircraft, is extremely time-consuming and costly. Qualification testing involves the material characterization of the fibre reinforcement, the polymer matrix resin, the uncured (intermediate) prepreg material, and the cured composite laminate. Material characterization consists of testing for the chemical, electrical, thermal, and various other useful engineering properties.



(Source: *Advanced Materials and Processes*, February 1991)

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Schematic of components in fatigue-testing machine



(Source: *ASTM Standardization News*, March 1993)

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## 5. TRENDS

### Test programmes zero in on P/M steels, formability

A growing number of manufacturers have begun to rely on advanced methods of materials characterization to identify the optimum performance envelopes for both processing and in-service conditions. Concurrent Technologies Corp. (CTC), 1450 Scalp Ave., Johnstown, PA, USA 15904, Tel.: 814/269-2456, Fax: 814/266-5106, is involved in several characterization projects that will impact the materials community. The work is funded by the US Navy's Manufacturing Technology Program through its National Center for Excellence in Metalworking Technology (NCEMT).

**Powder metallurgy:** Seven new powder metallurgy (P/M) steel systems are being characterized using the conventional property tests outlined in MPIF (Metal Powder Industries Federation, Princeton, NJ) Standard 35. They are: air-hardening steel, diffusion-alloyed steels, pre-alloyed steel with elemental nickel addition, pre-alloyed Mo-Mn steel, stainless steels, powder-forged steels, and MnS-treated steels. The data will be used to establish new P/M standards.

Other physical and mechanical properties also will be determined for these and current standard alloys. Tests include wear, fatigue, surface endurance limit, machinability, thermal expansion, shear/torsion, fracture toughness, hardenability, and corrosion resistance.

**Formability:** CTC's *Atlas of Formability* will quantify the materials processing characteristics of conventional and advanced materials. Data are being developed using a range of test equipment, including:

- A deformation testing system capable of evaluating intermetallic and metal-matrix composites, superalloys, and ceramics at deformation rates of 0.025 to 760 mm/s (0.001 to 30 in./s) and temperatures to 2,500° C (4,500° F);
- A high-speed mechanical compression tester that can simulate forging at deformation rates as high as 12.7 m/s (500 in./s), temperatures to 1,500° C (2,700° F), and loads to 450 kN (100,000 lbf);
- A hydraulic sheet-metal tester that can generate punch speeds up to 380 mm/s (15 in./s).

The atlas currently contains data on flow stress, forming efficiency, workability, and forming limits for several alloys, including Monel K500 (Inco Alloys International Inc., Huntington, W.Va., UNS N05500), aluminium alloys 6061 and 7050, TiAl AD intermetallic-

matrix composite (Martin Marietta Corp., Bethesda, MD), chromium steel, and alloys 600 and 625.

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### Robotic tensile testing saves time, boosts quality

During the 1990s, many manufacturing facilities and laboratories will adopt automated robotic tensile testing systems, which will frequently tie into company-wide local area networks (LANs). Robotic systems, because of their greater consistency compared with a human operator, can greatly increase productivity in large-volume, repetitive testing applications. When used in LANs, the computer-based systems can respond rapidly to the testing needs of different locations. Other positive influences on the demand for robotic testing include the growing need for more extensive data for statistical process control and other quality-related programmes, and decreasing prices for programmable robots. Robotic testing installations to date include multiple systems at a major aluminium manufacturing plant, and individual systems at several plastics research facilities.

In a typical system, a computer-controlled robot moves the specimen from a rack to a measuring station at which contact gauges automatically measure specimen width and thickness at single or multiple locations and a laser scans a bar code that contains test parameters. Next, the robot inserts the specimen into the grips of the tensile tester, the extensometer lowers onto the specimen, and the automated test begins. During testing, the robot extracts another specimen from the rack, moves it to the measuring station, and then returns to the grips to remove the broken pieces from the previous test. (Tinius Olsen Testing Machine Co. Inc., PO Box 429, Willow Grove, PA 19090-0429, USA, Tel.: 215/675-7100, Fax: 215/441-0899)

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### Automated Brinell testing meets productivity goals

Automated and semi-automated Brinell hardness testing systems will be widely used in the future because they are much faster than the conventional method of making test indentations manually and then measuring them with a hand-held microscope. Automated systems automatically position the indenter over the surface to be tested, move it to the indent position, and apply the 3,000 kg test load via a standard 10 mm carbide ball. The system's miniature TV camera then furnishes data on the edges of the test impression to a microprocessor, which calculates the average diameter based on edge differential-contrast (rather than the impression depth). The diameter, hardness value, and an enlarged image of the impression are displayed with 2 s on the computer



monitor. The indenting step is manual in semi-automated systems.

Automated Brinell testing systems are in operation at Lukens Steel Co., Conshohocken, PA., and Taylor Wharton Co., Harrisburg, Pa., with another due to be installed at NRC Corp., Newton, Mass. Semiautomated systems have been installed at the Chrysler Corp. plant in Mexico City, Mexico, and at Bethlehem Steel Co.'s Steelton, PA., plant. (Tinius Olsen Testing

Machine Co. Inc., PO Box 429, Willow Grove, PA 19090-0429, USA, Tel.: 215/675-7100, Fax: 215/441-0899)

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(The source of the last three articles comes from *Advanced Materials & Processes*, January 1993)

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## 6. STUDIES, REPORTS AND PUBLICATIONS

### Critical Survey of Non-destructive Testing Techniques for Non-conducting Materials

ERA Report 92-0109R, June 1992

This 110-page report is a user guide to the less well documented non-destructive testing NDT techniques applicable to the newer materials, such as particle and fibre composites and ceramics, referred to here as non-conducting materials.

The report is divided into six sections, each covering one topic. Because of the wide range of applications not every use of the NDT techniques described has been dealt with but sufficient information is provided to allow interpolation and extrapolation of data for use on other applications. The growing use of computers to extend the range of tests to items previously thought untestable and to make the data user friendly is highlighted. (ERA Technology Ltd., Cleeve Rd., Leatherhead, Surrey KT22 7SA, U.K. Fax: 0372-3744-96)

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### Materials Testing for the Metal Forming Industry

By Klaus Pöhlant, Vienna: Springer-Verlag, 1989  
ISBN 3-540-50651-9, \$86.50 from D.A. Books

The testing of bulk and sheet metals for their suitability for forming operations represents a significant effort for both manufacturers of metal components and producers of semi-finished products. *Materials Testing for the Metal Forming Industry* is concerned with such test methods and is addressed to both researchers and engineers involved in the metal forming industry.

This small volume consists of seven chapters of double spaced text (plus five appendices and an index). The first six are a revised translation of the author's *Werkstoffprüfung für die Umformtechnik* (MRE, Springer-Verlag, 1986). Following the introductory chapter, the book progresses logically from the determination of stress-strain curves for bulk (Chapter 2) and sheet (Chapter 3) metal, to a very brief chapter on the application and applicability of flow curve tests to particular metal forming processes. Chapter 5 deals with formability limit testing for both bulk and sheet forming, and Chapter 6 with properties of the workpiece after forming. The scope of this English translation is broadened by the addition of Chapter 7 covering the testing of tool materials for bulk metal forming.

The content is very much concerned with the behaviour of the material under test, and brief descriptions of test procedures, with little emphasis on the testing equipment itself or detailed procedures. The

book should not be regarded as an introductory or instructive text, but rather as notes on the subject and a guide to the literature. It makes no pretensions of being a complete or comprehensive survey of the subject area indicated by the title. At times this brevity is a little frustrating considering the intended audience. An inspection of the references quickly reveals a strong bias towards the German literature, although attempts have been made to anglicize the book by incorporating references to ASTM test methods and replacing some of the German references with references to similar work from the English literature. Readers with a quantitative interest are catered for with a large number of equations, but these do not intrude on the readability of the work.

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### Tensile Testing

Edited by P. Han, 1992, 300 pp.  
ISBN 087170-440-4

- Learn ways to predict material behaviour through tensile testing;
- Learn how to test metals, alloys, composites, ceramics and plastics to determine strength, ductility and elastic/plastic deformation;
- A must for laboratory managers, technicians, materials and design engineers and students involved with uniaxial tensile testing.

Includes chapters on tensile testing of metals and alloys, plastics, elastomers, ceramics and composites, each written by an expert in that subject-matter.

Tensile Testing begins with an introduction and overview of the test, with clear explanations of how materials properties are determined from test results. Subsequent sections illustrate how knowledge gained through tensile tests, such as tension properties to predict the behaviour, including strength, ductility, elastic or plastic deformation, tensile and yield strengths, have resulted in improvements in materials applications. The text also illustrates instances where tensile testing has been used to improve products by predicting stresses and strains under different temperatures and loads.

From materials to be tested to the methods and equipment used, Tensile Testing is a comprehensive collection of the world's intelligence on the subject, a valuable resource for those in testing laboratories and for those conducting research in universities and government laboratories. List price £65.00, member price £49.00. ASM International, Materials Park, Ohio 44073, USA, Fax: (216) 338-4634.

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Application of Automation Technology to Fatigue and Fracture Testing

Edited by A. A. Braun, N. E. Ashbaugh, and F. M. Smith

American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. 296 pp., hard cover, 1990

Continuing advances of computer and software technology have allowed for an automation of materials testing systems and processes to become significant. Though an initially expensive and complicated accessory to a materials testing system, automation has, in recent years, grown and reached a stage of being both inexpensive and a necessary subsystem. Several test techniques now desire having the speed, consistency and computational capabilities inherent in these systems. Over the years, rapid advances in software technology have facilitated shorter application development time coupled with higher application performance. Advances in software technology have kept pace with decreasing hardware costs thus providing the software which is essential for both testing system control and for data acquisition.

The book is divided into three major sections that deal with latest developments in the areas of implementation, analysis and management of laboratory test systems.

- Section I: System Implementation
- Section II: Analysis and Simulation
- Section III: Laboratory Systems and Information Management

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Ultrasonic Testing of Materials

4th, fully revised edition  
By J. Krautkramer and H. Krautkramer  
Springer-Verlag, 1990  
ISBN 3-540-51231-4, \$182.50

Divided into four categories, it covers the physical principles of ultrasonic testing in part A. This includes the motion of ultrasonic waves, their behaviour within materials and at interfaces, and their generation and detection by piezo-electric materials. Also included are some of the newer techniques for generation and detection of ultrasound including EMATs and lasers.

Part B covers instrumentation, an explanation of its operation and limitations, and various techniques used for assessing material integrity and thickness. It covers digital thickness measurement and points out some of the reasons for incorrect readings and how to avoid or overcome them. Methods of imaging, including variations of B and C scans and many others, are outlined, as is ultrasonic microscopy and acoustic holography. It very briefly touches on acoustic emission,

which is referred to as Sound Emission Analysis (SEA), but would have been better ignoring this area altogether.

Part C covers general ultrasonic testing techniques, including coupling (sometimes overlooked by inexperienced operators), the effect of real boundaries on the ultrasonic signal and defect detection and sizing.

Special test problems, i.e. application of ultrasonics to real inspections, are covered in Part D. Applications include forgings, rail, plate, sheet, strip, billets, wire, pipes, castings, weldments and fibre composites. Specific problems associated with a wide range of materials are well covered in Chapters 29 and 30.

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Non-destructive Characterization of Composite Media

R. A. Kline  
Technomic Publishing AG, Basel (1992)  
193 pp., soft cover (English), SFr. 143

A typical non-destructive evaluation of a composite material usually tests to find large-scale defects and provides little information about the nature of the defect, its effect on mechanical properties of the material, and the expected performance of the part. The book examines the features of ultrasonic wave propagation that can yield important quantitative information about composite materials, their microstructure, and their mechanical properties. This guide to NDE through acoustic wave propagation details both theory and practical applications. The book includes: advanced ultrasound methods for detailed identification and measurement of defects, and characterization of microstructure and mechanical properties. The contents of the book include: Basic Governing Equations, Wave Surfaces, Energy Propagation, Bulk Wave Propagation Anisotropic Media, Guided Waves, Experimental Consideration for Ultrasonic Measurements, Methods for Elastic Modulus Reconstruction for Ultrasonic Data, Experimental Considerations for Dynamic Modulus Measurement in Anisotropic Media: Phase Velocity vs. Group Velocity, Ultrasonic Modulus Measurements in Composite Media, and Composite Microstructure Characterization.

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Non-destructive Testing

This report describes the state-of-the-art of the principal NDT techniques, reviews current R&D activities within companies and research institutes, examines applications by industry sector and analyses the world market for NDT equipment. A directory of research laboratories, corporate players and associations is appended. The world market for NDT equipment is currently estimated at \$800 million per annum shared roughly 30 per cent by Western Europe, 30 per cent by the USA and 40 per cent by the rest of the world. This market is likely to grow at 5-10 per cent p.a. over the

next decade as western industry continues to modernize and automate, and as industrialization progresses in the developing countries. *Contents:* Executive Summary; Introduction; NDT Techniques, Applications; Research and Development Activities, The Market for NDT Equipment; Appendices list Research Laboratories; Manufacturers and Professional and Trade Associations. Innovation 128, 170 pp. 10/88, \$480 (£300). (World Business Publications Ltd., 4th Floor, Britannia House, 760 High Road, London N12 8RY. Fax: 081-446-3659)

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#### Acoustic Emission Testing of Aerial Devices and Associated Equipment used in the Utility Industries

presents case histories and the benefits realized by utilities that have used acoustic emission testing of aerial equipment. Fifteen years of experience are included in 90 pages of peer-reviewed papers. Three of the featured papers are: Acoustic Emission Monitoring and Destructive Testing of Glass Fibre Reinforced Plastic Bucket Truck Booms; Personnel Qualifications and Certification for Acoustic Emission Testing of Aerial Devices; and Advances in AE Technology for Testing of Aerial Devices. The book is useful for utility fleet managers, test engineers, and manufacturers of aerial lift and digger derrick equipment. (List price \$21, member price \$19).

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Wear Testing of Advanced Materials addresses special problems engineers encounter when developing and using wear tests for ceramics and other advanced materials. The volume gives the reader a good overview of the more common tests currently used to characterize advanced materials. Eleven peer-reviewed papers from international experts discuss: wear test methods for advanced materials, specifically related to different applications; and analysis and interpretation of results from wear tests. The publication is intended for mechanical engineers, lubrication engineers, tribologists, researchers in friction and wear, and bearing designers. (List price \$45, member price \$41). (ASTM, 1916 Race Street, Philadelphia, PA 19103, USA)

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#### New Methods for Corrosion Testing of Aluminum Alloys

V. S. Agarwala and G. M. Ugiansky (Eds.) ASTM, 1991, 228 pp. (STP 1134, \$65, ASTM members \$52)

These 13 peer-reviewed papers were presented at the International Symposium on Corrosion Testing of Aluminum Alloys. Methods covered include those for assessing exfoliation corrosion, microbiological-influenced corrosion, and stress corrosion. (ASTM, 1916 Race St., Philadelphia, PA 19103-1187, USA; Tel.: 215/209-5585; Fax: 215/977-9679)

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#### Non-destructive Test Equipment

Eight-page brochure from Magnetic Analysis Corp., Mt. Vernon, NY, USA describes instruments and systems for non-destructive evaluation of materials and components. Included is information on eddy current and ultrasonic testing equipment, instruments to detect flux leakage from magnetic materials, and a variety of accessories such as automated parts-handling systems.

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#### Fastener Testing

Eight-page brochure from Testing Instruments Div., NewAge Industries Inc., Willow Grove, PA, USA, describes measuring systems for torque/tension measurement of threaded fasteners, and direct-reading microhardness testing instruments for decarburization measurement of threaded fasteners. The fully automated microhardness testing systems can conduct decarburization tests in less than two minutes.

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#### Quality Control Software

Eight-page brochure from Perkin-Elmer Corp., Norwalk, CT, USA, describes QC Expert software for quality control of inductively coupled plasma (ICP) spectrometry experiments. The software allows users to create and automate their own testing quality protocols, allows unattended operation, and provides customized reporting and data analysis functions.

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#### Laboratory and test furnaces

Tube, split-tube, box, split-box, and a variety of high-temperature furnaces for laboratory and materials-testing applications are featured in Bulletin 3110/a (36 pp.) from Applied Test Systems Inc., Butler, PA, USA. Also described are reports, controlled-atmosphere chambers, and temperature controllers and recorders.

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#### Multipurpose vacuum furnace

The Model M60 is a multipurpose, front-access furnace for vacuum or controlled-atmosphere operation at temperatures to 2,000° C (3,630° F). Applications listed in a four-page brochure from Centorr Furnaces, Suncook, NH, USA include brazing and heat treating. An optional kit adapts the furnace to mechanical-testing machines. Included are water-cooled, stainless-steel pull rods.

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### Alloy for extensometers

The properties of Haynes alloy No. 214 are detailed in Brochure H-3008B (24 pp.). Haynes International Inc., Kokomo, IN, USA says the nickel-base alloy's excellent resistance to oxidation makes it suitable for use as extensometers for tensile and creep-rupture testing at temperatures to 1,095° C (2,000° F).

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### Ceramic-testing systems

Self-aligning grips, special fixtures, furnaces, capacitive extensometers, and other products support bend testing of ceramics to 1,000° C (1,830° F) and tensile testing to 1,600° C (2,910° F). Complete information is provided by Instron Corp., Canton, MA, USA, in "Ceramics Testing Systems and Accessories" (Bulletin WB-1005A, 12 pp.).

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### Accessory-selection guide

"Tools for Testing at Every Temperature" is a 20-page guide to grips and fixtures, extensometers, furnaces, and other mechanical-testing accessories for applications from -130 to 2,000° C (-200 to 3,630° F). MTS Systems Corp., Minneapolis, MN, USA, has sectionalized the brochure by test temperature.

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### Universal testing machine

A computerized Electromatic IV universal testing machine equipped with an environmental chamber is being used for climbing-drum peel tests of honeycomb-core panels at various temperatures. The electromechanical tester is described by Tinius Olsen Testing Machine Co. Inc., Willow Grove, PA, USA, in Bulletin 110-A, 12 pp.

### Non-contacting extensometer

The Vision Extensometer is an alternative to strain-gauge, clip-on extensometry, says Sintech Div., MTS Systems Corp., Stoughton, MA, USA, in Brochure 10/14. The non-contacting device uses a high-resolution camera to provide digital strain data that are compatible with computerized testing systems. One use: strain measurement inside an environmental chamber.

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### Environmental chambers

Bulletin 70 (4 pp.) from United Calibration Corp., Huntington Beach, CA, USA, describes a line of

standard environmental chambers for tensile and compression testing at temperatures from -75 to 540° C (-100 to 1,000° F). All models are heated by electrical-resistance elements. Digital controls maintain temperature within  $\pm 1^\circ$  C ( $\pm 2^\circ$  F). The company also builds custom, high-temperature test furnaces.

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### Testing composites, ceramics

Solutions to problems in mechanical testing of composites and industrial ceramics are explored by Zwick of America Inc., East Windsor, CT, USA, in Application-Information Bulletin AI 12 (8 pp.). Illustrated are custom systems for high-temperature testing of multidirectional laminates, silicon nitride, and silicon carbide.

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### Basics of laser extensometry

Strain in specimens heated to temperatures as high as 2,500° C (5,000° F) can be accurately measured using a laser extensometer, states Zygo Corp., Middlefield, CT, USA. An Application Brief titled "Laser Extensometer Testing" (Bulletin SB-0203, 4 pp.) and a brochure on the 1100 Series laser extensometer (Bulletin SB-0215B, 4 pp.) have details.

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### Aluminium-plating technology

Billed as a pollution-free alternative to cadmium plating, Ivadizing (ion vapour deposition of aluminium) operates at temperatures below 120° C (250° F). A literature package from Abar Ipsen Industries, Feasterville, PA, USA, details the coating process, and its use in applications such as fasteners, fittings, gas-turbine parts, and airframe components.

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### Densifying aluminium nitride

Technical Support Bulletins ANS-1 and ANS 2 cover: methods of processing aluminium nitride: forming green bodies by dry pressing of powder, and densifying green bodies by pressureless sintering. They are available from Advanced Refractory Technologies Inc., Buffalo, NY, USA, a manufacturer of aluminium nitride and other advanced ceramics.

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### Submerged-arc welding

Wires and fluxes for submerged-arc welding are described in a new handbook from Alloy Rods Corp., Hanover, PA, USA. Included are AWS electrode

designations, electrode-selection guidelines, suggested welding parameters, and troubleshooting tips.

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Aluminium-alloy properties

The 1990 edition of the Aluminum Association's popular "Standards and Data" reference contains composition limits, mechanical and physical properties, comparative characteristics, and other useful technical information for commonly available aluminium alloys and product forms. Price of the 220-page book is \$25 (Aluminum Association members, \$12.50). Copies may be ordered by telephoning the association's Publications Dept. (Washington, D.C.) at 301/645-0756.

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Aluminium-extrusions manual

A joint publication of the Aluminum Extruders Council and the Aluminum Association, the 130-page Aluminum Extrusion Manual covers the process, alloys and tempers, the competition, applications and designs, dies and shapes, finishes and tolerances. The single-copy price is \$34. Order from: Aluminum Extruders Council, 1000 N. Rand Rd., Suite 214, Wauconda, IL 60084, USA; Tel: 708/526-2010.

Ultrasonic-assembly guides

Five technical-information bulletins on ultrasonic assembly (16 pages total) have been prepared by Branson Ultrasonics Corp., Danbury, CT., USA.

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The following five books can be obtained through: ASTM European Office, 27/29 Knowl Piece, Wilbury Way, Hitchin, Herts, SG4 OSX, England. Tel.: 0462-437933, Fax: 0462-433678, Telex: 325684.

1. Composite Materials: Testing and Design  
(10th volume)

Editor: Glenn C. Grimes, Lockheed Advanced Development Co.  
495 pp. (1992); hard cover  
List price: £85.00; member price: £77.00  
ISBN 0-8031-1426-5

Important composite materials technical issues are discussed in eight sections:

- Compression Test Methodology Analysis and Development;
- General Test Methodology Analysis and Development;

- Material Mechanical Properties and Failure Criteria;
- Advanced Materials Analysis and Test;
- Analysis, Test, and Certification of Structure;
- Quality Assurance and Process Control;
- Interlaminar Fracture Analysis and Test;
- Damage, Flaws and Repair.

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2. Metal Matrix Composites: Testing, Analysis, and Failure Modes

Editor: W. S. Johnson  
300 pp. (1989), hard cover  
List: £56.00; ASTM member: £45.00  
ISBN 0-8031-1270-X

Reviews continuous fibre reinforced metal matrix composites (MMCs). Fourteen peer-reviewed papers on testing techniques, analysis approaches, and descriptions of various failure processes. For aerospace, academia, material research laboratories; those involved with mechanically characterizing MMC.

3. Elastic-Plastic Fracture Test Methods: The User's Experience (2nd volume)

Editor: J. A. Joyce

This new publication from ASTM presents papers from experts who share their practical experiences with the testing of elastic-plastic and fully plastic materials.

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4. Rapid Load Fracture Testing

Editors: Ravinder Chona, Texas A&M University, William R. Corwin, Oak Ridge National Laboratory  
192 pp. (April 1992), soft cover  
List price: £51.00; member price: £46.00  
ISBN: 0-8031-1429-X

This new volume from ASTM reviews the state-of-the-art of rapid loading to determine the fracture toughness behaviour of ferritic steels in the ductile-to-brittle transition region. In particular, papers focus on test methods that could:

- Reduce the amount of data scatter;
- Illustrate or establish any relationships between  $K_{Ic}$ ,  $K_{Ia}$ , and/or  $K_{Ia}$ ;
- Provide lower-bound measures of fracture toughness; and
- Improve the efficiency of testing with material of limited availability.

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5. Composite materials: fatigue and fracture  
(3rd edition)

Editor: T. K. O'Brien  
848 pp. (1991), hard cover  
List price: £120.00; member price: £96.00

This new volume from ASTM presents the state-of-the-art in testing and analysis of fatigue and low velocity impact of composite materials. Thirty-eight papers by authors from universities, government, and industry give a balanced presentation on these subjects:

- Matrix Cracking and Delamination;
- Interlaminar Fracture Toughness;
- Delamination Analysis;
- Strength and Impact;
- Fatigue and Fracture.

For aircraft designers, researchers in aerospace materials, composite material manufacturers, aerospace professors; also engineering designers, engineering professors.

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Composite materials: testing and design (ninth volume)

List price \$94.00; member price \$75.20

Focuses on significant advances in the area of damage tolerance and durability of composite structures. The book contains 27 papers divided into sections on Structural Considerations and Analysis, Delamination Initiation and Growth Analysis, Damage Mechanisms and Test Procedures, and Other Test and Design Subjects. (ASTM = American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103, USA)

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Hardness testing

Technical information from New Age Industries Inc., Willow Grove, PA, USA, describes micro- and light-load hardness testers designed for case depth analysis. The instruments can provide a profile of effective case depth to 50 HRC in less than two minutes. Manual or microprocessor-controlled models with loads of 500, 1,000 or 5,000 g (17.5, 35, 175 oz) are available.

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Composites testing

Four-page brochure from Micromet Instruments Inc., Newton Centre, MA, USA, describes the ICAM-1200/QC automated dielectric testing station for analysis

of flow, viscosity, and curing properties of thermosetting materials. The instrument is suitable for use with advanced composites, resins, foams, adhesives, sheet and bulk moulding compounds, and epoxy moulding materials.

\*\*\*\*\*

Ultrasonic testing

Six-page brochure from Ultrason Laboratories Inc., Boalsburg, PA, USA, details non-destructive testing products and services. It includes information on ultrasonic transducers for a variety of applications, as well as on microstructural, interfacial, dimensional, and mechanical testing and analysis services.

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1991 ASTM Directory of Testing Laboratories

List price: \$65; member price: \$52  
ISBN 0-8031-1243-2

This volume contains listings of more than 1400 laboratories. Most of them are located in the United States; more than 60 are located in Canada; still others are located throughout the world. The directory contains extensive indexes, making it easy to search by location, laboratory name, fields of testing, materials, and/or products. (ASTM, 1916 Race Street, Philadelphia, PA, 19103-1187, USA)

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Eddy current testing

Catalogue, 24 pages, from Foerster Instruments Inc., Pittsburgh, PA, USA, provides information on eddy current test instruments for non-destructive evaluation of materials and components. Included is information on specific applications, such as in-line testing of steel bar products, testing of semi-finished products for transverse and longitudinal defects, and measurement of tube wall thickness.

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Fastener testing systems

Literature from MTS Systems Corp., Minneapolis, MN, USA, includes information on systems for hatch testing of high-performance aircraft fasteners. The equipment combines standard load frames with special fixturing and control software for high-throughput fatigue testing to applicable military specifications.

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## 7. PAST EVENTS AND FUTURE MEETINGS

### 1993

- 12-14 May  
London  
Total Quality in the Steel Industry  
Sponsored and organized by the Ironmaking and Steelmaking Committee, Institute of Materials, 1 Carlton House Terrace, London SW1Y 5DB, UK. Fax: 071 839 3576
- 7-11 June  
Turtle Bay,  
Oahu, Hawaii  
6th International Symposium on Nondestructive Characterization of Materials  
(Center for NDE, 102 Maryland Hall, The Johns Hopkins University, Baltimore, MD 21218, USA)
- 13-15 September  
Washington, DC  
Forum on Materials and the Global Environment  
(ASM International, Materials Park, Ohio 44073-0002, USA. Fax: 216 338 4634)
- 15-17 September  
Beijing, China  
4th International Seminar "Environmentally and Energy Efficient Heat Treatment Technologies"  
(Heat Treatment Institution of CMES, PO Box 907, Beijing, 100083, PR China)
- 19-22 September  
Vancouver, BC,  
Canada  
2nd International Conference on Structural Applications of Mechanical Alloying  
(ASM International, Materials Park, Ohio 44073-0002, USA. Fax: 216 338 4634)
- 1-5 November  
Ballarat, Victoria,  
Australia  
INTERFACES 2  
An International Conference on the Structure and Properties of Interfaces and their Role in Advanced Materials Design, Processing and Performance  
(Centre for Advanced Materials Technology, Department of Materials Engineering, Monash University, Clayton, Victoria 3168, Australia. Fax: 61 3 565 4998)
- 15-19 November  
Los Angeles, Calif.  
19th ISTFA (International Symposium for Testing and Failure Analysis)  
(ASM International, Materials Park, Ohio 44073-0002, USA. Fax: 216 338 4634)
- 16-17 November  
Fort Worth, Texas  
Bolted and Bonded Joints in Composite Materials - Symposium  
(More information: Boeing Commercial Airplane Company, PO Box 3707, Seattle, WA 98174-2207. Fax: 206 662 2934)
- 6-7 December  
London  
Metal Matrix Composites 4 - Design and Innovation  
(Institute of Materials, 1 Carlton House Terrace, London SW1Y 5DB, UK. Fax: 071 823 1638)

### 1994

- 16-17 May  
Montreal, Canada  
12th Symposium on Composite Materials: Testing and Design  
(Symposium Chairman, Northrup Corporation, 1 Northrup Avenue, Hawthorne, California 90250. Fax: (310) 332 1481)



Advances in Materials Technology: Monitor

Reader Survey

The *Advances in Materials Technology: Monitor* has now been published since 1983. Although its mailing list is continuously updated as new requests for inclusion are received and changes of address are made as soon as notifications of such changes are received, I would be grateful if readers could reconfirm their interest in receiving this *Monitor*. Kindly, therefore, answer the questions below and mail this form to: Ms. A. Mannoia, Technology Development and Promotion Division, UNIDO, P.O. Box 300, A-1400 Vienna, Austria.

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Computer access number of mailing list (see address label):

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Position/title:

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1. Is the present address as indicated on the address label correct?
2. Do you wish to continue receiving issues of the *Advances in Materials Technology: Monitor*?
3. Which section in the *Monitor* is of particular interest to you?
4. Which additional subjects would you suggest to be included?
5. Would you like to see any sections deleted?
6. Have you access to some/most of the journals from which the information contained in the *Monitor* is drawn?
7. Is your copy of the *Monitor* passed on to friends/colleagues, etc.? If so, how many?
8. Do you have any information/suggestions etc. you would like to pass on to other readers?
9. Do you wish to have a specific "material" covered in a future *Monitor*?
10. Do you wish to contribute to the compilation of a future issue of the *Monitor*, be it with the main article or other information related to the relevant subject?
11. Please make any other comments or suggestions for improving the quality and usefulness of this *Monitor*.

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