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EMERGING
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SERIES

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New and Advanced Materials

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EMERGING TECHNOLOGY SERIES:

NEW AND ADVANCED MATERIALS

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CONTENTS

UNIDO's *Emerging Technology Series: New and Advanced Materials* is established as a mechanism of current awareness to monitor developments in the materials technology sector, and inform governments, industry and academia, primarily in developing countries.

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Dear Readers,

Under the leadership of the new Director-General of UNIDO, who took office on 8 December 1997, UNIDO was restructured in line with the Business Plan approved by Member States. Based on this Plan, a new programmatic approach for the Organization was developed, with integrated services designed to support sustainable industrial development and to help recipient countries solve their problems in the rapidly changing global economic environment.

Ten packages of services that encompass the following areas of activities will constitute the core of future UNIDO activities for both the public and private sectors: Industrial Governance; Industrial Export Capacity-Building; Industrial Information Networking; International Industrial Partnerships; Quality and Certification for Industrial Competitiveness; Cleaner Production and Environment; Efficient Energy Development; Agro-Related Industries Development; Local Industrial Development; and Women Entrepreneurship Developent.

For industrial growth and competitiveness, it is widely recognized that new materials technology has a crucial enabling role. Ever-increasing pressure on all sectors of manufacturing industry to reduce cost, and at the same time improve quality and reliability of products, means that materials with predictable performance are in great demand. Hence, testing and evaluation of modern materials is an important underpinning activity for industry and the cost of such work can be quite substantial.

In order to respond to the demand from developing countries in building-up/strengthening their technological capacity in this vital area, UNIDO is carrying out the preparatory and pilot activities phase for the establishment of the International Centre for Materials Evaluation Technology (ICMET) whose purpose is to develop international guidelines, codes of practice, standards on testing and characterization of new materials as well as seeking for cooperation with other international, regional and national organizations, industrial and professional societies. ICMET also runs training courses on advanced techniques in the area of materials science and engineering.

Since materials science and engineering (MSE) is a major tool for a wide range of industrial sectors which will have a major influence on economic and industrial competitiveness, UNIDO will establish the International Materials Assessment and Application Centre (IMAAC) in 1998. This centre will deal with techno-economic issues of MSE and will serve the countries to efficiently utilize their raw and natural materials resources for industrial application, taking environmental and energy issues into consideration.

We hope that our readers will be interested in UNIDO's programme in the area of new materials and we will be pleased to provide them with detailed information on request.

Vladimir Kozharnovich
Programme Coordinator

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CONTENTS *(cont'd)*

A. SPECIAL ARTICLE

ADVANCES AND TRENDS IN MATERIALS MEASUREMENT AND EVALUATION METHODS

by

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1. Introduction

For industrial growth and competitiveness, it is widely recognized that advanced materials technology has a crucial enabling role. Ever-increasing pressure on all sectors of manufacturing industry to reduce cost, and at the same time improve quality and reliability of products, means that materials with predictable performance are in demand. Hence, materials properties information from validated test and characterization methods, based on an understanding of the microstructure and processing parameters, is needed for improved materials selection, design and manufacture.

Testing and evaluation of modern materials is thus an important underpinning activity for industry and the cost of such work can be quite substantial. Advances are being made continuously in the development of new techniques, instrumentation, data analysis and predictive methods for assessing materials performance. In this article some of the general advances and future trends in materials testing and measurement are discussed.

Key areas of interest are the following:

(a) **Modelling.** Models describing materials behaviour based on an understanding of the microstructure, physical and chemical mechanisms, and process characteristics are being increasingly used to reduce cost and to increase reliability of testing;

(b) **Intelligent testing**. With the availability of rapidly advancing computing power, more intelligence is being built into the test system so that

- Tests are becoming more automated with increased throughput; this includes automatic test-piece preparation, dimensional measurements of complex shapes and finish;
- Test machines are interfaced to sophisticated control and analysis systems allowing complex testing to be undertaken in a rapidly assured manner;
- Real-time analysis and modelling can be undertaken;
- Powerful feedback loops can be used in real time to control the test;
- Sensors to monitor parameters such as loads, temperature, dimensions, and state of sample can be used effectively.

In this general area, tremendous progress is being made possible through the development of advanced control instrumentation incorporating sophisticated IT systems.

(c) **Prediction of long-term performance**. There is an increasing demand for life prediction methods. Thus, for example, lack of reliable methods for assessing life or durability has been identified as a major barrier for the introduction of composites into structural applications. Durability has so far been addressed mainly on an empirical basis with the result that large quantities of test data are available but are of limited use. To make real progress, the basic mechanisms of degradation need to be understood and used to establish test methods and predictive models. Research is under way for the development of test methods, including accelerated testing, to estimate the relative durability of composites in order to facilitate materials selection. Short-term tests that predict reliably the long-term behaviour of materials and systems in service are also being developed in conjunction with modelling work.

(d) **Process related measurements**. Over the last ten years, it has been widely accepted that materials processing holds the key to successful exploitation of many modern materials. Performance is intimately linked with process parameters and therefore much effort is being placed on test and measurement of materials properties for input to process models. These models are vital for the successful manufacture of industrial products using materials that can now be tailored to meet specific performance requirements.

(e) **Advanced characterization techniques**. Microstructure has a vital influence on materials performance and therefore, advanced techniques covering atomic microscopy, trace chemical analysis, digital imaging, nanoscale analysis and testing are having a significant impact in materials measurement and testing.

(f) **Miniaturization of testing**. There is a trend towards the need for testing small samples of materials because certain materials are mostly produced in small quantities or are thin e.g. coatings and oxides, thin rolled sheet, splat cooled materials or samples taken from components in service. Miniaturization can also be used sometimes to replace a large and expensive conventional testing system possibly with increased flexibility in mechanical, thermal and environmental test conditions. Examples of systems include a miniaturized thermal fatigue rig, a miniature tensile testing, a creep measurement technique for small disc samples and a miniature wear testing system.

(g) **Traceability, standards and reference materials.** Emphasis on traceability of measurements, determination of uncertainties and the use of standards and reference materials has increased notably in recent years.

With globalization of industry and trade, international harmonization of test method standardization is becoming common. Adoption of quality systems incorporating formal accreditation of tests and laboratory operations is also gathering pace at a fast rate.

In the following sections, some of the above aspects are discussed in more detail. It is not intended to provide a comprehensive cover of all aspects of materials testing and evaluation. Instead, examples have been selected to cover some salient points.

2. *Process-related measurements*

Increasingly, industry is aiming to achieve improved product quality through process control rather than end-of-line testing. Hence there is a growing emphasis on in-line monitoring of processes linked to real-time control. Process routes are frequently expensive, as can be raw materials, so the use of process models is becoming increasingly critical for competitiveness. Not only do we need good process models, but we must also have good materials properties data as inputs to the models. The quality of data has a direct impact on the output.

2.1 Measurements for processability of liquid metals

Physically based models to describe industrially important processes such as casting, spray forming, welding, primary metals production and secondary refining are now a very important part of modern production routes involving liquid metals. A key limitation to the successful introduction of these models is the lack of thermophysical data such as: enthalpy of fusion, specific heat, thermal conductivity, viscosity, density and surface tension.[1]

It is generally assumed that the materials properties and their temperature dependence is known, but this is often not the case. The relationship between the types of fault found in typical castings, the physical models required and the thermophysical properties inherent in the models are shown in table 1. In recent years, methods have been developed at NPL, for measuring many of the necessary thermophysical properties for casting of commercial alloys. These methods are now able to provide materials property data, for a range of industrial alloys at temperatures up to 1,600 °C.

Table 2 summarizes the techniques developed at NPL to measure a wide range of physical properties for liquid metals and alloys. These techniques have been found to provide satisfactory results. However, problems are encountered with some of these techniques when applied to light metals as a result of (i) the mechanical strength of the oxide skin formed by aluminium alloys and (ii) the tendency of magnesium alloys to vaporize at temperatures around the melting point and (iii) the general reactivity of both metals in the liquid state. Work is currently in progress to take account of these factors.

Table 1. Models and relevant thermophysical properties for metal casting

Physical process	Thermophysical data required	Prediction
Solidification and heat transfer	Density vs temperature Specific heat vs. temperature Conductivity vs. temperature Latent heat of fusion Emissivity (metal/mould, furnace wall) Liquidus temperature Solidus temperature Interface heat transfer coefficient	Hot spots Effectiveness of riser Effectiveness of chill Effectiveness of insulation Solidification direction Solidification shrinkage Microporosity
Micro-modelling	Phase diagram Chemical species composition Solid fraction vs temperature Number of nuclei per volume Growth constant for eutectic Diffusivity of solute in solvent Gibbs Thompson coefficient	Microstructure morphology Grain size Grain orientation
Fluid flow	Viscosity vs. temperature Surface tension Coefficient of friction-metal/mould	Cold shut Mould filling time Effectiveness of ingate Effectiveness of runner Pouring rate Pouring temperature

Table 2. Current capabilities in measuring high temperature thermophysical properties for materials processing

Property	Temperature range (K)	Method	Apparatus and/or probe	Materials	Uncertainty (%)
Density	300-1,900	Archimedean	BN or coated metal bob	Potentially most materials	±1
		Hydrostatic probe			
	1,300-2,300	Levitated drop		Metals	±2
Viscosity	300-1,870	Rotating cylinder viscosity	Mo or Pt bobs and crucibles	Slags, glasses, salts, MMCs	±10
	300-1,870	Oscillating viscometer	Under construction	Metals, salts	
Surface tension	1,300-2,300	Levitated drop	Fourier analysis of oscillation spectrum	Metals	±2
	300-1,900	Hydrostatic probe	BN or coated metal probe	Potentially, most materials	±10
		Cylinder detachment			
Heat capacity	200-1,000	DSC	Perkin-Elmer	All	±1
	1,000-1,800	High-temperature DSC	Polymer laboratories	All	
Enthalpy	1,000-1,850	Drop calorimeter	PRT & AC bridge	All	±1
	1,300-3,000	Levitated drop calorimeter	Under construction	Metals, alloys	±5
Thermal conductivity	300-1,500	AC line source method	AC	Salts, slags	<±5
Thermal diffusivity	300-1,500	Coated-line source method	Under development	Metals	
Electrical conductivity	300-1,870	4-electrode method	Under development	Slags, salts, metals	
Thermal expansion	150-1,700	Mechanical dilatometer	Silica apparatus to 1,000 k, alumina at high temperature	All; small components at low temperature	Typically ±2
Thermal stability	300-1,600	Thermo gravimetry	Platinum apparatus	All	Typically ±5

2.2 Prediction tools

Measurements mentioned above are time consuming and there is frequently a need to estimate physical properties. At NPL, two different types of model are available to calculate properties of alloys from a knowledge of the chemical composition. MTDATA[2] is a thermodynamic model used to calculate phase equilibrium and thermodynamic properties such as the nature of phases formed, melting and transformation temperatures, heat capacity, enthalpy and fraction solid, mainly for equilibrium conditions, but with some capability to introduce kinetic effects. Comparison between the predicted and experimental values gives confidence for the results of particular classes of alloys both in the experimental technique and in the predictive approach. Particular advantages of the predictive approach are: (a) calculations are fast and reliable once the relevant data are available and (b) calculations can be used to explore the change in properties across the specification of a given alloy. MTDATA can be linked with fluid dynamic software to model micro and macro segregation associated with the casting of a specific component. Combination with more sophisticated diffusion modelling permits calculations to be carried out that takes into account differences in cooling rates.

METALS MODEL,[3] the second predictive tool, is a less accurate technique but can be used to estimate values for the following physical properties with the uncertainty shown in brackets: heat capacity, enthalpy (<5 per cent), thermal conductivity (solid 10 per cent, liquid 25 per cent), viscosity (20 per cent) and density (<5 per cent).

2.3 Friction measurement in metalworking

In metalworking, friction has a major influence on the yield and quality of the final product. Models are being used to optimize the process conditions and reduce or eliminate defects in the finished product. The success of these modelling techniques depends heavily on the reliability of the data on materials behaviour, in particular the friction generated between the deforming metal workpiece and the tool. Data currently available using instrumented systems are inadequate due to large errors, so the metalworking contact conditions are now being simulated in miniaturized test systems which enable values of friction under well controlled contact conditions to be measured.

The most important consideration is that in many metalworking processes the tool is cold and the workpiece is hot. In one of these miniaturized systems,[4] the workpiece sample is a thin strip of the metal under test which is heated under computer control by a DC current in a specially designed sample holder (figure 1). The DC self-heated workpiece sample replaces the pin in a conventional pin-on-disc test system. The frictional and normal forces, and the relative displacement in the friction and normal directions are all recorded continuously by computer.

In pilot experiments using an AISI 52100 bearing steel as a simulated tool material, and mild steel as the hot workpiece material, friction coefficients in the range of 0.30-0.45 have been measured in tests where the initial workpiece temperature was up to 1,000°C (figure 2).

2.4 Plastics processing

Injection moulding is a widely used technique for plastics processing and involves some interesting developments associated with process-related measurements.

Commercial software packages already exist which model the injection moulding process (e.g. Moldflow, C-Mould and Fillicalc). Use of these packages is increasing to improve product reliability and to reduce time to market for new products. The materials testing requirements to support these packages are also likely to increase due to the fact that property data representative of real industrial situations are needed. For example, pressure can increase viscosity by 500 per cent and yet over 99 per cent of modelling work uses data collected at normal pressure without correction. Another example is the effect of cooling rate. Industrial processes operate at high speed, but much of the property data have been collected at or near equilibrium conditions. This difference can have a significant effect on shrinkage and warpage of critical plastic mouldings.

Increased use of optimization is another trend. One approach to this is to capture as much information on the process in real time and then use an expert system to determine how to correct faults, how to set up the process in the first instance or to provide real-time feedback to keep the process within acceptable limits.

In recent years there have been major developments in catalysts that have led to a much greater control of molecular weight, branching and co-monomer placement in polymers. This gives opportunities to fine-tune the processing and subsequent material properties to a much greater extent than was possible previously. These many new grades of polymers can be expected to match customer requirements. This will increase the demand for more materials property data which are expensive to obtain. Therefore predictive methods that will enable industry to reduce the cost of data acquisition will be of significant interest.

Other processes like film blowing, thermoforming and rotational moulding, do not have the same level of software available at present, as is the case with injection moulding. Commercial software will no doubt become available in due course, accompanied by similar data requirements to those outlined above.

3. *Mechanical testing*

Progress in mechanical testing of materials has been rapid with the availability of advanced sensors, instrumentation and computers. Developments in experimental methods, data acquisition and data processing are assisting industry to keep pace with the demands for the testing of new materials and their characterization under increasingly severe loading and environmental conditions.

Design and analysis of structural components both require knowledge of loading and environmental conditions, component shape and materials properties. The basic design goals of safety and economic competitiveness have been expanded to include functional performance, durability, aesthetics and reliability. Mechanical testing plays a key role in the design process and provides information on:

(a) Materials response such as non-linear, visco-elastic, anisotropic behaviour;

(b) Stress analysis obtained from material stress-strain relationships;

(c) Failure behaviour through identification of fracture mechanisms and modes; and

(d) Life-prediction through accelerated testing.

Most mechanical testing is carried out under monotonic tensile loading. However, there is an increasing need to carry out more complex tests to determine the material and structure response under in-service conditions. Compression and shear properties are particularly important for composite materials, but the methods vary and consensus on the best test is not widespread.

Demand for biaxial and multiaxial tests is becoming increasingly evident. Thin walled tubular specimens under axial loading, internal pressure and torque are used, but test methods need further development. There is indeed a great lack of biaxial data. Servo-hydraulic tension-torsion testing mechanics that allow independent control of the loading components are available, but specimen preparation and design of fixtures for local stress introduction are expensive.

There is considerable activity in high-rate and impact testing of materials. These require data acquisition at microsecond levels, which poses extreme challenges for sensors and recording systems. Simultaneous recording of several strain gauges and real-time monitoring of fracture under impact are on the increase. Special, often expensive, methods have been developed for material characterization at high strain rates, up to a few thousand strain units per second, but these generally cater for very specific needs.

Dynamic fracture behaviour of brittle materials is very important in understanding and preventing catastrophic failure of components. This can be investigated by new hybrid analytical/experimental approaches using high-speed digital recording implementation and high-speed photographic equipment.

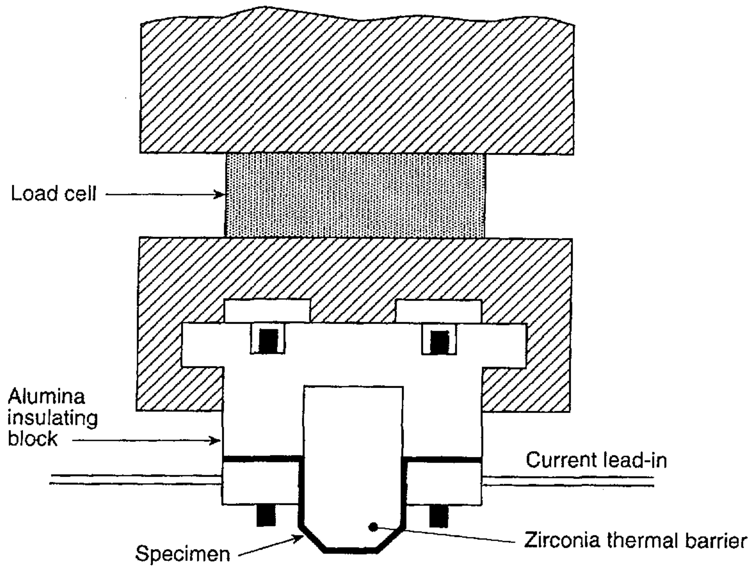

Figure 1. Schematic diagram of DC heated pin system

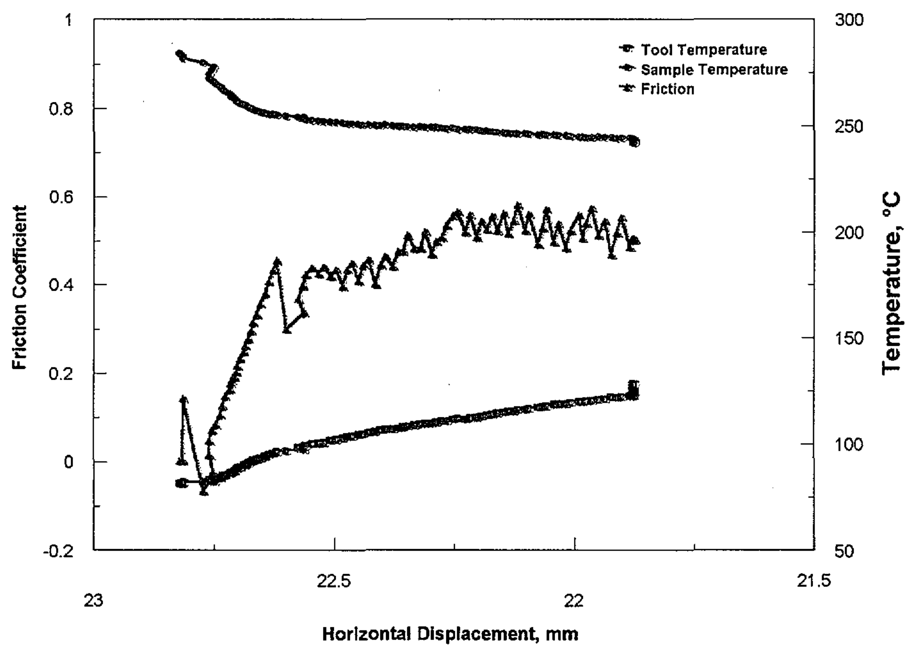

Figure 2. Results of friction measurement at initial workpiece temperature of 300° C

For studies on damage accumulation and life prediction, there is a growth of computer-controlled multi-parameter fatigue testing combined with intermittent or real-time non-destructive evaluation. Spectrum fatigue loading controlled by computers for simulating expected in-service conditions is being used to determine the feasibility and validity of accelerated testing for life prediction.

For improving efficiency, as in power generation systems, materials able to withstand increasingly higher temperatures are being developed. Consequently the need for development of facilities capable of operating at very high temperatures is growing consistently. Thus the development of high-temperature composite materials, such as ceramic matrix composites, provide real challenges for testing. A suitable test system must include compatible frame, grips, furnace and extensometers. The latter are particularly important. Optical, electro-optical and laser extensometers require further development. Similarly, innovative test methods are often coupled with innovative test specimen and fixture design. Methods are needed for determining the stress-strain behaviour to failure of the material in tension, compression and shear, and also for the measurement of modulus, strength, ultimate strain and fracture toughness as a function of temperature.

In mechanical testing, advances are also being made in the design of test specimens, grips for specimens and load introduction systems by using modelling and particularly FEA techniques. It is possible to predict regions of high tensile stress that could eventually lead to premature and invalid failure. This approach can considerably reduce the time required to develop a new test method and at the same time increase confidence in the results.

The extension use of IT and the sophistication of modern test machines enable processing of test data to be carried out fast and automatically. This "black-box" type approach has to be treated with care. As an example, we can consider processing by the test machine of tensile test data. Perfectly linear elastic responses are rare, yet the system will typically compute a value for Young's Modulus. This can be done in a variety of ways, e.g. the elastic curve may be linearized mathematically, a tangent may be taken—but where on the curve? A secant modulus may be calculated, but between which points?

Widely different results may be obtained, particularly when curves have a high slope, as for metal and ceramic matrix composites, so it is essential that the user is fully aware of the underlying mathematics used by the software in the matrix in order to be able to fully understand the property values that are being generated.

4. *Virtual testing*

Composite materials are being used increasingly in innovative design and products. They are attractive because they can be tailored to meet performance requirements that cannot be obtained using single/monolithic materials. Thus, by changing the fibre, the volume fraction of the fibre, the matrix or the way the fibres are incorporated into the matrix, materials with improved resistance to thermal distortion or stiffness to match operational requirements can be produced. Although the flexibility of the system is a real advantage, it would be an expensive and time consuming task to experimentally test every possible combination of fibres and matrix material. Recent progress in modelling computer techniques offers new and relatively inexpensive tools that allow designers to understand and analyse the properties of components far more easily. The use of mathematical simulations of materials behaviour to predict property data can be described as virtual testing. With advances in mathematical modelling and the availability of increasing computer power at affordable prices, this type of testing will become much more common. Of greatest value are models based on physical principles rather than those derived from empirical data fitting procedures, for it is only in a soundly based model that one can have real confidence. Of course, empirical models are very useful, but their limitations must be kept in mind.

Some modelling techniques, finite element analysis (FEA), for example, are complex and demand expensive input on the part of the user. In FEA, first a grid of elements is produced to represent the component and the model works out the behaviour of the material inside each element. These individual calculations are then aggregated to describe the behaviour of the material of the component as a whole. However, the FEA method becomes very expensive as soon as these input parameters are changed, e.g. in order to take account of progressive damage and the nature of the change. However, by studying what happens in the composites at the microstructural level, researchers have developed models that can forecast the likely characteristic of particular fibre/matrix composites based on the properties of the fibre, the matrix and the interface. These micro-mech-anical models can reliably predict the initiation and growth of damage in single-ply and in multiple-ply laminates.

Recent work at the National Physical Laboratory in the UK has produced a demonstrator that shows the potential of the virtual testing concept for the special case of composites. Reliability of predictions is crucially linked with the quality and efficiency of the models. The mathematical models used in this case have been developed at NPL.[5] Starting from fibre and matrix properties, predictions can be made of progressive ply crack formation during loading by applying energy principles that lead to lower band or safe performance levels. The model can be used to calculate mechanical properties of a laminate as the stress is increased, for either uniaxial, biaxial or shear leading modes. The degradation of the material property with accumulated ply cracking is calculated by these programs, thus allowing the engineer to predict the likely non-linear behaviour of the material.

A graphical interface using virtual reality has been developed for ease of selection and change of input parameters giving a very informative representation of the laminate and its subsequent damage and the resultant properties. When a laminated composite material is tested, in tension for example, the first mode of damage is the formation of ply cracks in the matrix between adjacent fibres in plies aligned perpendicular to the principal tensile stress. As the load increases more ply cracks will form, followed eventually by fibre breakage in the plies having fibres parallel to the principal tensile stress. Delamination between the plies can also occur with ultimate failure of the laminate. In the current NPL demonstrator, only the first mode of damage has been considered because ply cracking alone leads to the initial significant degradation of critical mechanical properties.

A simulation can take between 1-30 seconds depending on the complexity of the laminate and the sedation of the maximum crack density. Development of damage with increasing stress in the form of objects representing localized ply cracking can be displayed. Individual plies can be extracted from the laminate so that the pattern of cracks can be studied in more detail[6] (figure 3). Properties, such as "crack density vs. stress", can be calculated from the program (figure 4).

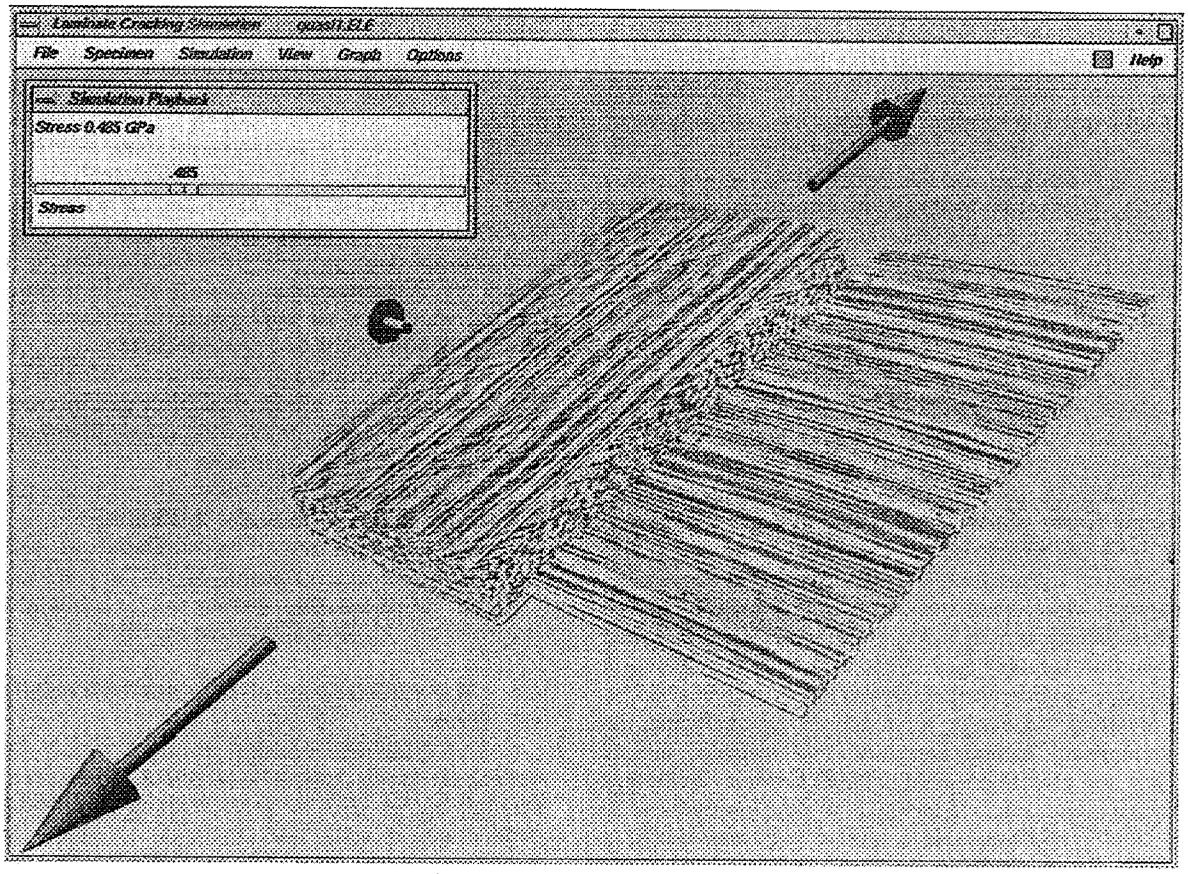

Figure 3. A picture from the virtual composite testing system showing cracks in a multi-ply system

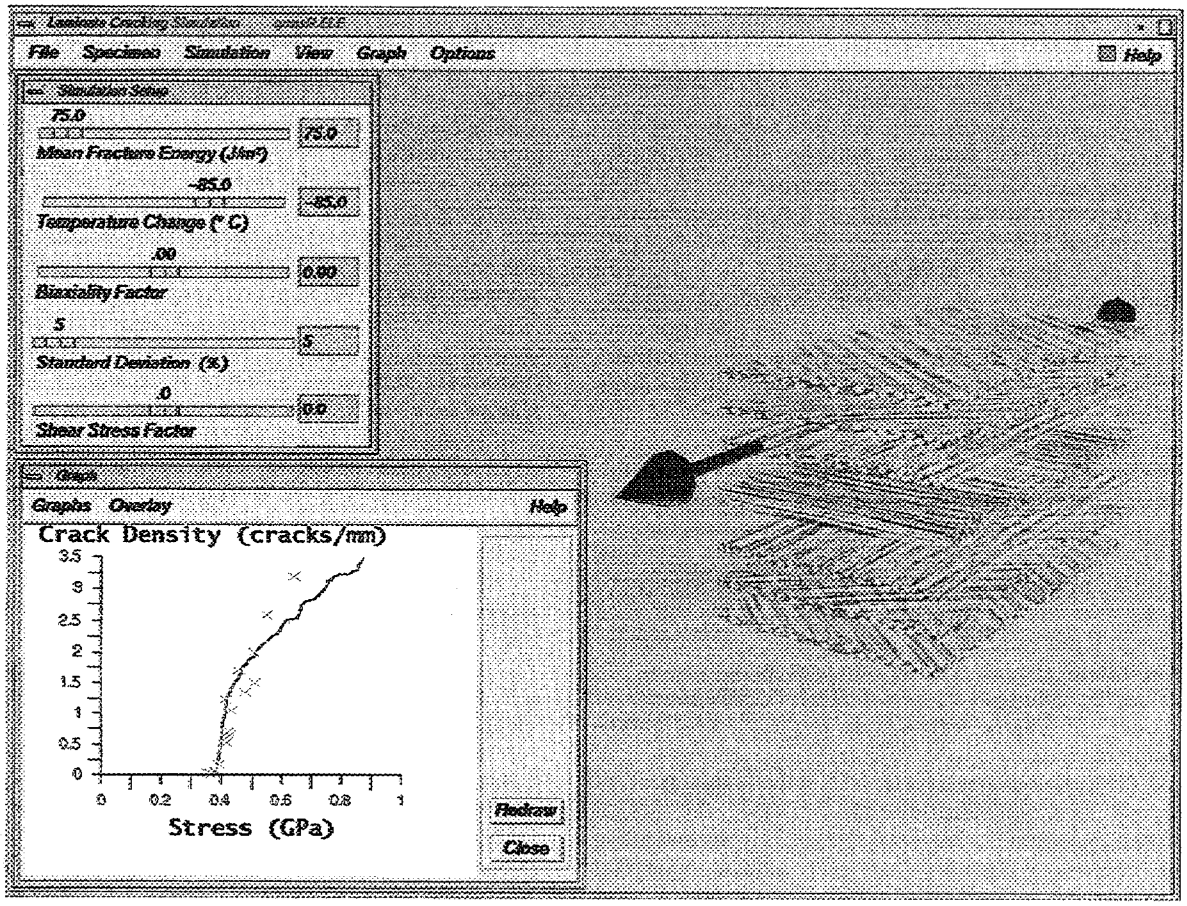

Figure 4. An example of the graphical output available from the virtual composite testing system

The models developed for ply cracking in composite laminates can easily be applied to other layered materials and systems. Applications exploiting both the mathematical models and the 3-D graphic interface have already been developed for the prediction of tensile cracking in oxide coatings and cracking in the epitaxial layers of semiconductors. These enable the prediction of cracking as a result of change in temperature and stress in the layered system. Virtual testing is thus a very powerful technique and it seems certain that the demand for this type of approach will grow steadily.

5. Surface analysis

Surfaces play a very important role in materials science and numerous spectroscopy techniques are available for chemical analysis of the surface or the near-surface regions. These techniques, such as Auger Electron Spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS), are widely used, the first to obtain elemental composition and some chemical information from the top few atomic layers of a material, while the second provides information on both elemental identity and chemical bonding such as functional groups and molecular types. XPS, also known as Electron Spectroscopy for Chemical Analysis (ESCA) can be used for depth profiling, valuable, for example, in analysing ion implementation, homogeneity of carbon-fibre surface chemistry and effectiveness of catalyst surfaces. In the last ten years a major advance has been to develop methods to obtain reliable and traceable quantitative measurements.[7,8] Therefore, results obtained for measurements carried out using different instruments anywhere in the world can now be compared on an absolute and quantitative basis.

Much of this type of work was carried out under the VAMAS programme[9] with the participation of many countries, and hence we can expect to see the methods to be adopted as international standards through ISO's Technical Committee 201 on Surface Chemical Analysis.

Following on from the AES and XPS activities, efforts are being made to develop reliable and qualitative measurement methods for secondary ion mass spectroscopy (SIMS). This involves the analysis of a mass spectrum of particles emitted from a surface when the surface is exposed to an energetic beam. It is an ultrasensitive technique for identifying molecules on a surface, as well as a depth profiling tool for detecting very low concentrations of contaminants or ion-implanted species. SIMS techniques include static SIMS (SSIMS), dynamic SIMS, time of flight SIMS (TOF SIMS), laser induced mass analysis (LIMA) and sputtered neutral mass spectroscopy (SNMS).

SSIMS can identify both organic and inorganic molecular species. A low flux of ions are bombarded on the sample revealing information about its outermost surface layer. Dynamic SIMS uses a much higher intensity bombarding beam and has a very low detection level (ppb) which is necessary for depth profiling of dopants and trace elements in semiconductors. It also can give x-y distribution maps of atomic species with sub-micron spatial resolution. Therefore, the technique can provide both lateral and vertical distribution of charge carriers in ion-implanted microelectronic devices.

As the technique has developed and the requirement for better resolution and sensitivity has increased, the time of flight mass spectrometer has been successfully introduced to assess complex molecules that have similar molecular weights but are comprised of different elements; a mass resolution of greater than 0.001 atomic mass unit can be achieved. Applications include analysis of thin-film molecular contamination in optical fibre or reflection coatings and multi-layer fibres deposited by molecular beam epitaxy method.

Table 3 gives a summary of some of the important techniques currently used for chemical analysis of surfaces with an indication of their scope and capabilities.

Due to rapid progress in fields such as communication medicine and aerospace, the need for absolute chemical and structural characterization of contaminants, dopants, implanted species at or near material surfaces is growing rapidly. Future trends and needs include:

(a) Identification of molecular absorbates and monolayers for polymers, tribological films and biomaterials;

(b) Analysis of multi-layer organic films composed of lipids, proteins and/or surfactants;

(c) Selective and non-destructive surface profiling to a depth of 1-5 μm in steps of 1 to 10 nm;

(d) Non-destructive measurement of defects and residual strains on a surface or within the material;

(e) Methods that enable the examination of biopolymers and biologically active organics.

6. Characterization of thin surface coatings and films

Thin films and coatings are used widely to improve optical, electronic, magnetic, aesthetic, wear, corrosion and thermal barrier performance. With rapid advances in the production of such thin coatings, the applications are becoming more demanding and the necessary test and measurement methods correspondingly difficult. It is recognized that the properties of nanometre to micrometre thick films are often different from their counterparts in bulk form leading to the development of new measurement techniques.

Thus, for topographic characterization, the conventional stylus type instrumentation may not be appropriate either because the resolution is not sufficiently high or because the coating may be deformed unacceptably by the measurement process itself. However, to obtain surface profiles at the atomic/molecular scale, one can use scanning tunnelling microscopy.

The integrity of the surface with the coating can only be sustained if its mechanical properties are compatible with the substrate over the range of conditions experienced in service. Measurement of these properties of thin films and coatings is now possible using a nano-indenter (figure 5). This instrument employs a differential capacitive technique to characterize thin films and coatings with force and displacement resolutions of 10 μN and 0.05nm-1 nm respectively, and submicron level spatial resolution. Quantitative hardness data can be obtained, but more importantly the load/displacement curve gives values of Young's modulus and information on coating adhesion.

The nano-indenter can be further enhanced by integrating a scanning force microscope (SFM) into the optical microscope part in place of a standard objective lens. The net advantage of combining surface topographic and indentation data in one instrument, with an accurate electromechanical positioning system, is that a particular sample site can be located and measured before repositioning either under an optical objective lens or the SFM. Lateral scan range of about 20 μm or more can be achieved. With such an instrument, measurement of the mechanical properties of multiphase materials with fine heterogeneous microstructure can be undertaken. For example, duplex stainless steels with a microstructure consisting of ferrite and

Table 3. Selected techniques for surface and near-surface chemical and elemental analysis

Technique	Surface, <10 nm	Near-surface <2 µm	Bulk, >10 µm	Depth profiling available	Used to identify interfaces	Quantitative	Elemental	Chemical	Used to identify organics	Used to identify inorganics
	No depth profiling									
AES/SAM	Yes	No	No	Yes	Yes	Yes	Yes	Some	No	Yes
EDX/WDX	No	Yes	No	No	Some	Yes	Yes	No	No	Yes
ESCA (XPS)	Yes	No	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Dynamic SIMS	Yes	No	No	Yes	Yes	Yes	Yes	Some	No	Yes
SSIMS	Yes	No	No	No	No	Some	Some	Yes	Some	Some
TOF SIMS	Yes	Some	No	Yes	Yes	Some	Yes	Yes	Yes	Yes
FTIR	No	Yes	Yes	No	Yes	Yes	No	Yes	Yes	Some
XRF	Some	Yes	Yes	No	Some	Yes	Yes	No	No	Yes

Source: Surface Science Laboratories, Division, Fisons Instruments, Mountain View, California.

Key to techniques

AES/SAM	Auger electron spectroscopy and scanning Auger microscopy
EDX/WDX	Energy-dispersive X-ray and wavelength-dispersive X-ray analyses
ESCA (XPS)	Electron spectroscopy for chemical analysis (or X-ray photoelectron spectroscopy)
Dynamic SIMS	Dynamic secondary-ion mass spectrometry
SSIMS	Static SIMS
TOF SIMS	Time-of-flight SIMS
FTIR	Fourier-transform infrared spectroscopy

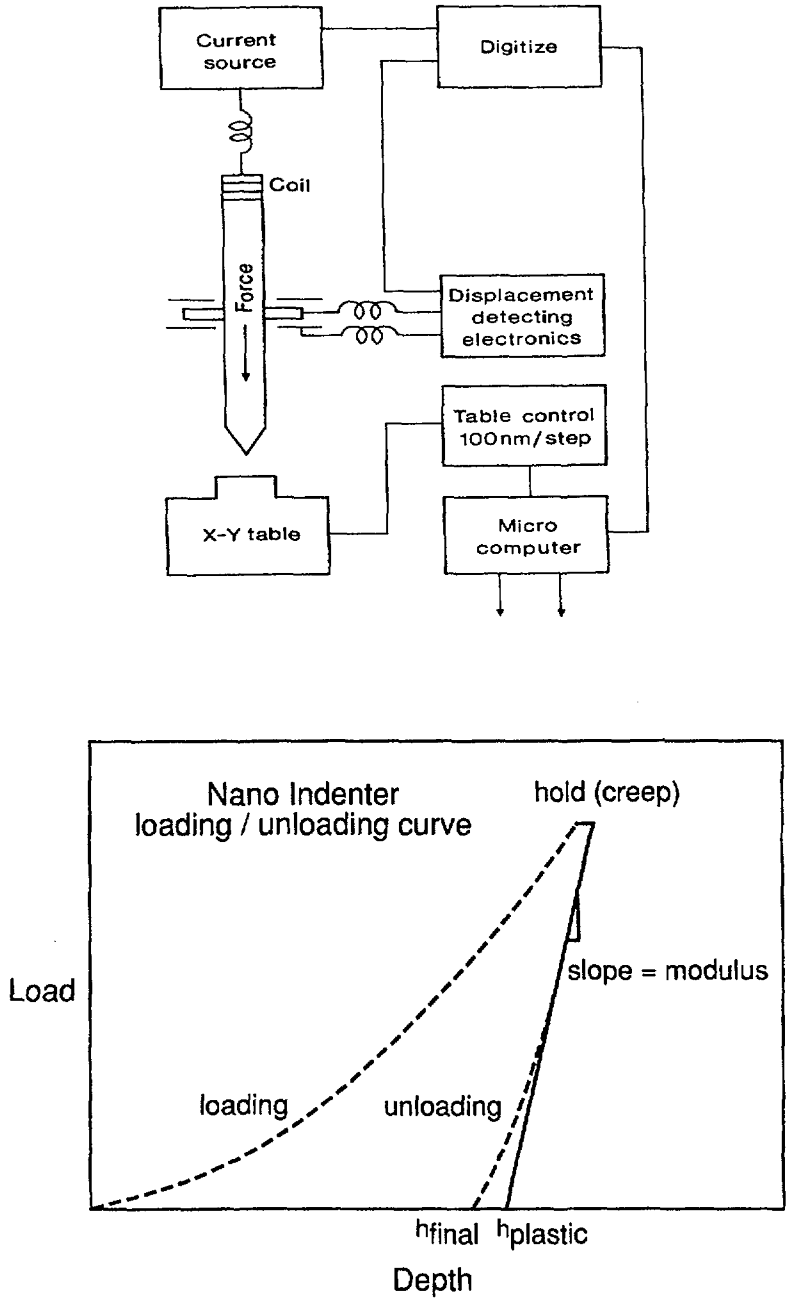

Figure 5. Schematic diagram of a nanoindenter and a load-displacement curve

austenite have been investigated[10] to establish their susceptibility to long-term ageing at intermediate temperatures (300-400°C).

7. Miniaturized test systems

In various areas of materials testing and evaluation, conventional systems have drawbacks. For example, the system can be large and expensive to purchase and operate; requirements for large specimens cannot be met where only small samples are available; and the range of environmental control can be limited due to cost and other practical constraints. Such difficulties may be overcome by innovative and miniaturized test methods and systems.

7.1 Thermomechanical testing

As an example, NPL has developed a new miniaturized electrothermomechanical test rig (ETMT) which is computer controlled and uses electrical resistance heating for the power source (figure 6). The system can be operated to study uniaxial and thermomechanical fatigue deformations and to obtain data on properties that change with temperature, such as modulus and thermal expansion. The rig has been used to measure and study phase transformations and electrical resistivity changes with varying temperatures in steels, Ti alloys and TiAl intermetallics. Also, thermal fatigue properties of hard tool materials have been measured together with yield in compression and tension at temperatures up to 1,330°C.[11] The versatility of the miniaturized system means that it can also be used to study the kinetics of microstructural changes and other thermal effects on a variety of materials.

7.2 Tribology testers

For abrasion testing, a miniaturized ball cratering test has been developed[12] and the system is expected to be available commercially. The cost is a small fraction of the more conventional tests and very small quantities of materials are required but the data can be comparable.

Similarly, a micro-tribology tester has been developed. Here, instead of measuring wear and friction on a large scale with large samples, the idea is to use a test system which replicates the individual events that occur in the contact between two surfaces, i.e. asperity contact.

8. Non-destructive evaluation

Conventionally, non-destructive testing of materials concentrates on the detection of macroscopic defects such as cracks in structures and devices in service. However, there is an increasing requirement for non-destructive characterization to be used innovatively in all aspects of materials production and application.

Examples of recent techniques include the use of ultrasonic velocity measurements for materials thickness, high-resolution tunnelling microscopy for detecting surface features, nuclear magnetic resonance and electron paramagnetic resonance for degradation of polymer-based composites and thermal wave imaging to detect delamination of ceramic coatings on metals. As in other areas of testing, sensitivity and detection levels are being increased continuously and developments are aimed at bringing sophisticated and highly technical equipment out of the laboratory and onto the shop floor.

In the X-ray analysis area, new X-ray sources, such as the synchrotron, together with improvements in the use of back-scatter, are potentially very powerful tools and should enable further expansion of X-ray techniques in materials evaluation. As an example, an evaluation system of the future (not a single piece of equipment) could combine several imaging modes involving X-ray, magnetic resonance, ultrasonics, and optical and thermal radiation. Thus a multi-mode analysis of a composite structure to detect a weak bond area could combine information from the X-ray source on a change in composition or on fibre-debonding, from the ultrasound source on the differences in strength in the region, and from the magnetic resonance source on the materials curing history.

Ultrasonics is one of the most commonly used techniques for non-destructive evaluation. Improved resolution, higher penetration power and higher frequencies in excess of 100 MHZ are increasingly available to allow detection of smaller imperfections. Ultrasonic techniques are now acceptable to test fragile or brittle materials, porous ceramics, metals and polymers, and liquid sensitive materials such as electronic substrates and superconductors. Even acoustically absorbent materials, such as graphite-epoxy composites, can now be examined using ultrasonics. Developments in higher frequency transducers are enabling resolution in the 20-30 µm region. With modern powerful computers the trend will be for greater availability of more automated and bench top systems.

Generally, applications for ultrasonics include evaluation of chemical composition, microstructure, internal stresses, cure, anisotropy, crystal structure, grain size and porosity. Developments in ultrasonic technology should increase the scope to the measurement of properties such as ultimate tensile stress, strain to failure, hardness, fracture toughness, thermal expansion, fatigue damage, and thermal and electrical conductivities.

9. Assessment of uncertainties in materials testing

Ever- increasing demand for high-performance products with greater reliability means that designers require estimations of uncertainties in test results. Indeed, ISO and many other prominent standards organizations have a policy 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 the tolerances specified in the relevant standard.

Two recent published documents are important in this context: (a) "Accuracy (trueness and precision) of measurement methods and results", (ISO5725) and (b) "Guide to the expression of uncertainty in measurement". The latter has been published jointly by leading authorities, namely ISO, IEC, IUPAC, BIPM, IUPAP and OIML.[13] This provides a comprehensive treatment based upon rigorous statistical methods for the summation of uncertainties from various sources but is complex for general use. Therefore, simplified versions have been produced, e.g. by the British Measurement and Testing Association.[14]

The total uncertainty of a measurement is determined by summing all the contributing components in an appropriate manner. So it is necessary to quantify all the components. For materials testing, a component smaller than one fifth of the largest component may be neglected. The ISO Guide categorizes two ways of evaluating uncertainties, A and B (figure 7). Type A is applicable when repeat observations can be made and a standard deviation for the measurements can be determined using conventional statistical analysis. Type B evaluation is by means other than for type A and makes use of, for example, tolerances specified in standards, measured data, calibration certificates, manufacturer's specification and in most cases a knowledge

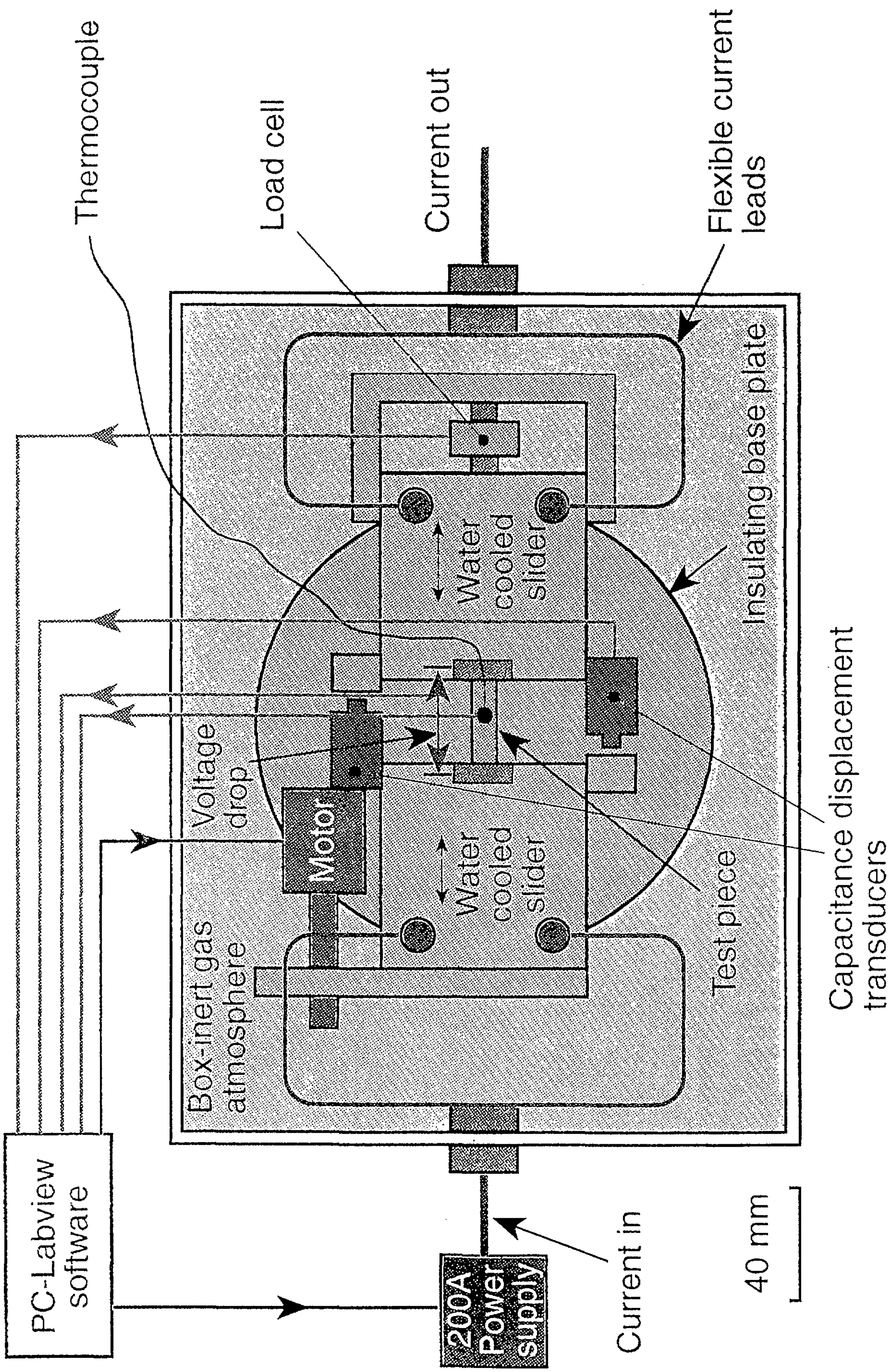

Figure 6. A miniaturized electro thermomechanical testing rig

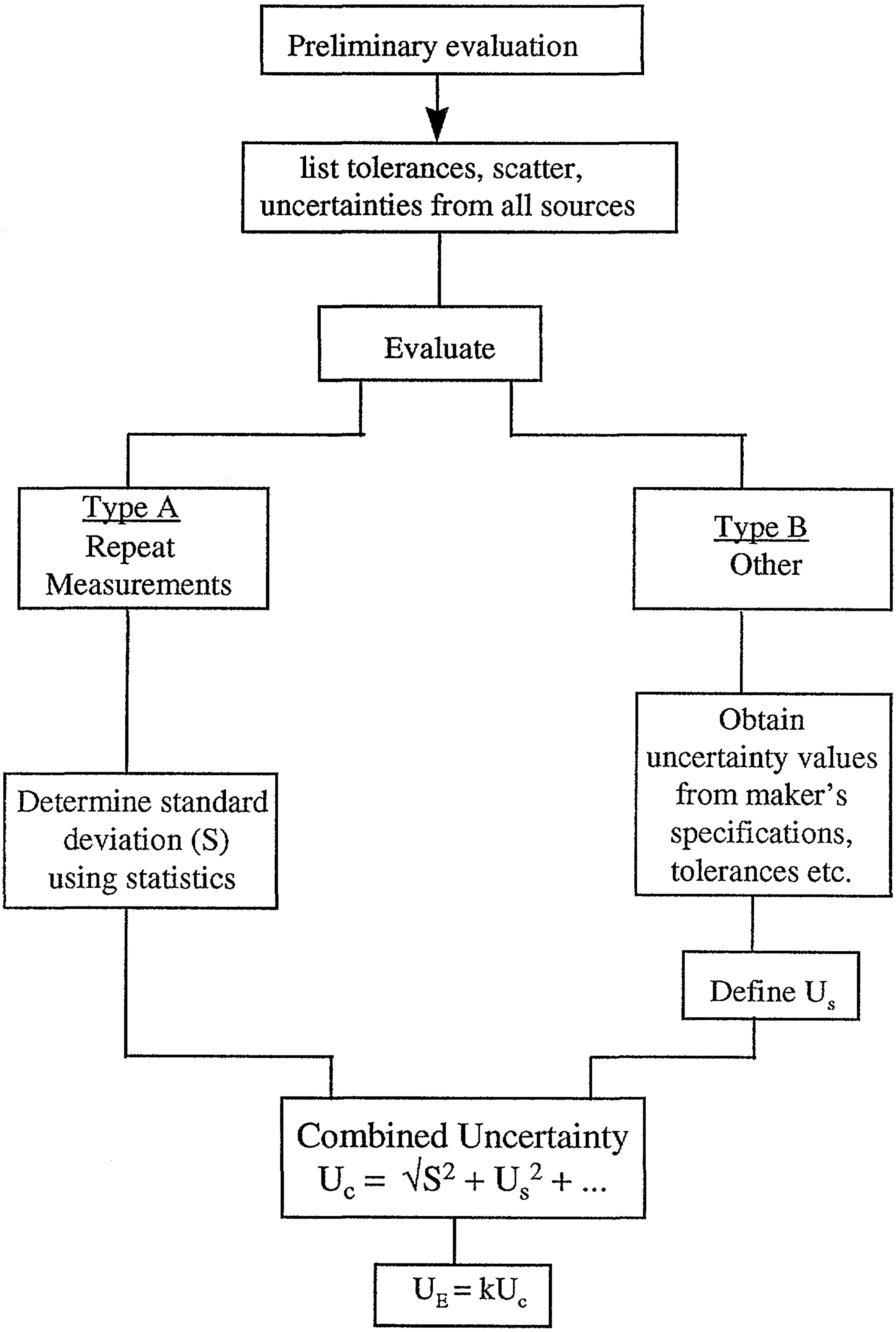

Figure 7. Outline method for calculation of uncertainties

of a simplified model of the relationship between the various components, and of the likely distribution model of the components. If, for example, the tolerances specified in a standard is ±a, then in the absence of any other knowledge, a rectangular distribution model may be assumed. The uncertainty is then $U_s = a\sqrt{3}$ where Us denotes a Standard Uncertainty obtained by multiplying U by a suitable factor.

The next step is to determine the Combined Standard Uncertainty, Uc, by summing the standard uncertainties, usually by using the root sum square method. Finally, the Expanded Uncertainty, U_E, is obtained by multiplying Uc by a coverage factor, k, where $k = 2$ for a 95 per cent confidence level and thus, $U_E = 2U_c$.

Using this approach, a method of estimating the uncertainty of measurement in uniaxial creep testing of metallic materials has been developed[15] and is proposed to be incorporated in the relevant European Standard.

Absolute statement of uncertainty can be made only when both material dependent and material independent components are known. Thus, for example, it is necessary to have a prior knowledge of the creep response of a material to temperature and stress before measurement uncertainty can be calculated.

Significant attention is already being paid by laboratories undertaking high-quality test and measurement work to the subject of measurement uncertainty and the trend is certainly towards an increase in emphasis.

10. International standards

For materials testing and standardization, a key issue is the lack of international standards. Although the value of global standards is generally recognized, the progress towards the establishment of such standards is slow and can take several years to achieve consensus for any particular standard.

Sometimes, even multinational companies apparently carry out the same test in different ways using different specimen size and test conditions in different operating countries. This means that results cannot be easily correlated or compared between test laboratories, and manufacturers have to duplicate efforts and indeed facilities. Adoption of the same standard would enable industry to gain competitiveness and product acceptance worldwide.

Over the last ten years, international standardization has indeed gathered momentum and both ISO and IEC have taken steps to accelerate the development of standards for rapidly developing technologies and to initiate significant new programmes. Increasing integration of economic and market activities in Europe is enabling greater cooperation between ISO, IEC and the European standardization bodies CEN and CENELEC. Rather than duplication of expensive and time-consuming standards development work, parallel voting of proposed standards is now taking place for European and international standards in many important areas.

A good example of how the international community is working together can be found in the area of surface chemical analysis. Certain basic surface analytical techniques (see section 5) are commonly used and although the principles are relatively well established, the operation of the instruments, their performance and the interpretation of the data can be complex.[7, 8] As a result, widely acceptable quantitative analyses and reliability can be difficult to achieve. Various national bodies like the ASTM in the USA, the Surface Analysis Society in Japan and the BSI in the United Kingdom have technical committees active in this field and some technical standards have been produced, notably about twenty by ASTM.

The ISO Technical Committee 201[16] on Surface Analysis was established about five years ago and has seven subcommittees: Terminology, General Procedures, Data Management and Treatment, Depth Profiling, Auger Electron Spectroscopy, Secondary Ion Mass Spectrometry, and X-ray photoelectron spectroscopy, with a total of 15 working groups. The coverage is fairly comprehensive. The Terminology subcommittee is concerned with the development of a conceptual framework for the terms used in surface analysis as well as standardization in the definition of terms. The General Procedures subcommittee develops standardized procedures relevant to two or more subcommittees, e.g. standardization of specimen preparation and handling and specification of reference materials. The Data Management and Treatment subcommittee develops standards for databases and for the transfer of data between instruments.

The remaining four subcommittees develop standards for the most commonly used surface analytical techniques—AES, XPS, and SIMS/SNMS and for depth profiling. They are concerned with standardization of methods for instrument specification, instrument operation, data acquisition, data processing, qualitative analysis and quantitative analysis. For example, instrument manufacturers currently specify the important parameters of their instrument in different ways making it difficult for users to obtain valid comparisons. Similarly, standards can assist analysts by giving guidance on specimen preparation and mounting, setting up of the instrument functions and experimental parameters. Standards for methods of identification of elements, chemical state and composition, and concentration are also of great value. Heavy dependence on computerized data processing and display for many instruments means standards are also required for specifying the properties of algorithms.

ISO TC has at last nine potential international standards in development and a further sixteen topics are under active consideration.

Last year ISO TC 201 made the following remarks about expected trends in surface chemical analysis and the expected need for further standards development:

"High spatial resolution (with parallel and normal to the surface) is required for the analysis of many industrial products and materials. Semiconductor devices, for example, are being produced with smaller dimensions. New classes of materials (e.g. composites) and devices (e.g. sensors) are being developed in which a surface or interface chemical composition needs to be controlled to meet various industrial needs. Nanotechnology, in which devices are fabricated on the nanometer scale, is of growing importance. Other industrial applications in which surface chemical analysis is now utilized, particularly in the polymers, ceramics, metallurgy, composites, adhesion, tribology, wear, corrosion, thin fibres and coatings, implant materials, catalysis, microelectronics, and superconductors sectors, continue to grow.

There is an increasing need for more detailed and more reliable quantitative surface analysis (for example, the identification of chemical phases present at surfaces and interfaces). There is also an increasing need for quantitative surface analysis with improved accuracy.

Commercial instruments for surface chemical analysis are now being supplied with increasingly powerful computer systems and software. As a result, there are needs to validate and extend new and existing algorithms used for the processing of measured data.

Recommended procedures, reference data and reference materials will be used to an increasing extent to ensure that surface chemical analysis can be made with the needed reliability and efficiency. International standards will also provide a convenient and efficient means of documenting conformance to quality management systems."

There is a great demand for standards in this area and in a survey of standards, Auger Electron Spectroscopy on its own revealed over 30 new potential topics.

11. Concluding remarks

Advances in materials measurement and evaluation techniques are taking place in parallel with developments in manufacturing technology. Steadily intensifying pressure on industry to reduce cost and the product innovation cycle, combined with improved reliability and quality of products, is leading to stringent, smaller scale, automated and intelligent testing based on modern IT systems.

The interplay between materials modelling and testing is becoming more important that ever before. Thus, models are used to accurately define the materials parameters which need measurement, to predict and simulate materials behaviour and testing, and to reduce the amount and cost of expensive test results.

In any test, it is essential to understand the measurement method and what the results actually mean. Property data which are ambiguous and poor in quality can have serious cost implications and can affect competitiveness of industrial products. Hence validated measurement techniques are in great demand. For high-quality testing of materials, traceability and uncertainty of measurements are also essential to establish.

Globalization of manufacturing and trade means such methods should be acceptable across national boundaries and preferably the measurement methods should be standardized internationally. It is encouraging to see that more international standards on advanced materials analysis and testing are beginning to emerge. International collaboration in harmonization of test methods through organizations like VAMAS has an increasingly important role to play.

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B. RECENT DEVELOPMENTS — NEW TECHNIQUES AND SYSTEMS

Dual-method coating thickness measurement

The new MP4 from Fischer Technology, Windsor, CT, offers a versatile, compact hand-held solution to coating thickness measurement. It combines the magnetic induction and eddy current methods of measurement, and is capable of measuring metallic and non-metallic coatings on steel and iron, and non-conductive coatings on non-ferrous base metals. The MP4 allows the user to interchange a wide variety of Fischer Smart Probes for different applications—a feature that allows the operator to cover diverse measurement applications simply by changing probes.

The MP4 features automatic probe recognition; microprocessor-control with digital LCD display; one point calibration and four point master calibration for different material conditions; seven application memories so measurements can be stored according to application; metric and USA unit indication; printer output with Cp and Cpk indexes; and a bi-directional RS-232 interface for downloading data to a computer, printer or data logger. A full statistical evaluation can be made on any measurement series. (Source: *Materials Evaluation*, September 1996)

Ultrasonic gated flaw detector

Xactex Corp., Pasco, WA, has introduced the XU-2250, an ultrasonic gated flaw detector with digital thickness gauge. High-speed operation is achieved through a number of techniques. The high pulse repetition rate (up to 10 kHz) enables the system to operate up to the maximum allowed by the ultrasonic test geometry. Multichannel systems are easily controlled with the XU-2250's advanced computer interface and control software. Sophisticated analog and digital signal processing techniques provide local data reduction, allowing for the acquisition and control of data from many more test channels.

Xactex includes DGC and AGC signal enhancement for thickness measurement. A programmable AGC allows the amplitude gate to measure the true signal amplitude, as the base gain does not change during testing. A DAC allows high-speed thickness testing by correcting for signal reduction as time of flight increases. (Source: *Materials Evaluation*, September 1996)

Thermal imaging systems

Cincinnati Electronics Corporation, Mason, OH, now offers specialized instrument lines for the steel and metals industries. These infrared imaging devices will find wide application in predictive and preventive maintenance and safety services across these processing industries.

Typical applications include periodic checking of ladles and torpedo cars, inspections of refractories, the location of faulty electric circuits and determining the heat patterns of furnaces. The available instrumentation for these applications ranges from the TVS-100 series of thermal video systems to the more sophisticated focal plane array system.

An optional software package compatible with Windows provides advanced analysis as well as standard and custom report generation. (Source: *Materials Evaluation*, September 1996)

316 stainless steel ID kit

The Alloy Detector 316 from Systems Scientific Laboratories, Walnut Creek, CA, identifies 316 stainless steel, differentiating between 316 and 304 stainless using the Electro-Lite alloy deposit system.

Less than 0.0001 mm (0.0004 in.) of metal is required by the system for virtually non-destructive results. An LED indicator light lets the operator know when the test is complete. The 316 kit, packaged in an attaché case, comes with enough materials for more than 1,000 tests. Replacement items are available from stock. (Source: *Materials Evaluation*, September 1996)

X-ray fluorescence spectrometer

Oxford Instruments, Concord, MA, has available an ED-2000 energy dispersive X-ray fluorescence spectrometer for the analysis of steel components. Up to 80 elements for qualitative analysis or up to 50 elements for full quantitative analysis between NA-11 and U-92 may be analysed simultaneously.

This digital technology system includes XpertEase software, based on Windows 3.1, along with Oxford's patented Pentafet detector and the latest in X-ray optics. The system can perform qualitative, full quantitative and semi-quantitative analysis of steel components even when such elements as aluminium, chromium, cobalt, copper, iron, manganese, nickel, phosphorous, silicon, sulphur, titanium and vanadium are measured together. (Source: *Materials Evaluation*, September 1996)

Real-time digital radiography system

MQS Inspection and Omega International, Elk Grove Village, IL, have joined to develop an on-line real-time radiography system for measuring pipe wall thickness. The ThruVu Inspection System, designed by Omega, allows MQS to offer fast, accurate inspection of both insulated and uninsulated piping while the pipe remains in service.

The ThruVu technique uses a low-intensity iridium-192 gamma ray source and a linear array of digital solid state detectors, positioned and manipulated by an automated, remotely controlled crawler. As the unit travels along the pipe, the detector array measures the intensity of the gamma rays after they have passed through the walls of the pipe. A portable computer continually collects and analyses the data, plotting actual thickness values instantaneously through Omega's proprietary interactive software. (Source: *Materials Evaluation*, September 1996)

Field instrument for X-ray fluorescence

The X-MET 960 from Metorex, Inc., Bend, OR, uses a custom solid-state microprocessor that has been miniaturized and dedicated for use in harsh field environments. Ruggedized hand-held X-ray fluorescence detector probes complete the system. Completely self-contained and battery-operated, the system and its convenient carrying case allow it to be carried and used by a single person.

Data collection, analysis, logging and management are automated. The system includes interactive software with simple pull-down menus to be used by the alloy analyser operator. Both qualitative and quantitative results are immediately available at the conclusion of the measurement. Interface ports are provided for probe, printer, external keyboard, external floppy disk and RS-232 serial data. (Source: *Materials Evaluation*, September 1996)

Alloy identification kit

Identify aluminium alloys in minutes with this new test kit from Koslow Scientific Co., Edgewater, NJ. The Aluminium Alloys ID Lab 1513 comes in a handy carrying case for use in the laboratory, scrap-yard, quality control, maintenance, repair, production and receiving areas. Alloy groups are separated based on quick tests for magnesium content, zinc, manganese, copper and magnesium silicide.

The ID Lab 1513 comes complete with all necessary supplies and easy-to-follow instructions. It can be used by personnel with no special training or background in chemistry or metallurgy. Rapid and clear colour spot development identifies elements in the aluminium alloy, classifying the alloy group. Single element tests take less than a minute to perform. (Source: *Materials Evaluation*, September 1996)

Wall thickness measurement

Cygnus Instruments, Inc., Annapolis, MD, has made available a completely revised Cygnus 1 Basic Model, designed to measure the thickness of metals without including the thickness of coatings. These gauges are typically used to measure the wall thickness of pipes, tanks and vessels, and will also measure on rough, corroded surfaces with minimal preparation (no grinding).

The Cygnus 1 Basic Model features an enlarged display, an operator selectable switch that toggles between metric and inch measurements, enhanced multiple echo measurement technique with a signal strength indicator, increased battery life and an automated low battery warning. (Source: *Materials Evaluation*, September 1996)

Advanced thermal imaging camera

Prism DS from Flir Systems Inc., Portland, OR, provides complete temperature data and analysis. Verify and quantify temperature data in the field or laboratory with the Prism DS hand-held thermal imager. Even small defects are revealed with its 320 x 244 high-resolution imaging. Save images to PC cards for documentation, later analysis, or to use in reports. A 12-bit dynamic IR range allows retrieval temperature data from any point on a stored image. Lens sizes from 8 mm to 10 mm; optical filters are also available. Measurement range: -10° to 1,500°C (14° to 2,700°F). Future system upgrades will be available on PC cards. Camera and lens weigh 2.9 kg (6.5 lb). Battery life: 6 hours. Ambient operating range: -10° to 45°C (14° to 113°F).

AnalyzIR post-processing software supports analysis of images captured by the Prism DS and other thermal imaging cameras. Capabilities include trending, area of interest analysis, delta-T measurements and multiple image analysis. Runs on Microsoft Windows 3.1+ and Windows 95. (Source: *Materials Evaluation*, October 1996)

PC-based ultrasonic systems

Krautkramer Branson, Lewistown, PA, features a revolutionary "all on one card per channel" solution for PC-based ultrasonic systems instruments. The new USPC2100 supports true parallel fire multichannel applications in automatic testing systems. Up to eight testing channels can be housed in either a standard desktop or industrial PC chassis. All ultrasonic and evaluation electronics for each channel are contained on a single PCI card that the user configures for either flaw detection or thickness measurement. Integration into new or existing installations is simple with easy to configure outputs.

Set-up and control of the system are fast and simple via a user-friendly Windows 95 operator interface. Particular attention has been given to the display of easily interpreted test results. The operator interface includes a strip chart recorder and a user-defined application screen that simultaneously displays results from multiple channels on a drawing of a test layout or component. (Source: *Materials Evaluation*, October 1996)

D Sight Aircraft Inspection System (DAIS)

Heeding the call of the commercial airline industry's need for affordably priced state-of-the-art NDI/E equipment, Diffractor Ltd., Windsor, Ontario, Canada, has advance engineered the D Sight Aircraft Inspection System (DAIS) to accommodate the hanger floor budget. DAIS is the optical technology that provides aircraft inspectors with visual data indicating severity levels of interlayer corrosion along fuselage lap seams. The fundamental operating principles of the trailblazing DAIS 250C are retained in this latest model. The full-featured DAIS 250C continues with a full graphical software suite based on a virtual diagram of an aircraft's surface for extensive image and data archiving, and remains the D Sight tool of choice for military fleet operators. The new battery-operated DAIS 250CB instantaneously displays D Sight images via on-board monitor or by optional head-mounted video display. Unlike ordinary visual methods, the DAIS 250CV brings a consistent expert eye to lap joint inspection procedure. DAIS technology allows all inspectors—regardless of their skill level—to immediately see comparable enhanced views of fuselage skin. (Source: *Materials Evaluation*, October 1996)

Portable video remote visual inspection

Olympus America, Inc., Industrial Products Group, has introduced two new, highly portable video remote visual inspection (RVI) combination units. The new units combine a built-in light source and appropriate video imaging equipment for videoimagescopes, flexible fibrescopes, or rigid borescopes in one compact package. Now a remote visual inspector in the plant and in the field can carry a scope, a light source, a video CCU, and a detachable LCD monitor in one unit, slung over the shoulder, and make a visual inspection in the interiors of machinery, turbines, gas or diesel engines, tubing, pipe assemblies, etc., with hands entirely free to operate the RVI equipment. (Source: *Materials Evaluation*, October 1996)

Fan-cooled, high intensity UV lamp

The Spectroline FC-100 fan-cooled UV lamp from Spectronics Corporation, Westbury, NY, produces super-high-intensity long-wave ultraviolet light. It is ideal for NDT, quality control, UV curing, weld testing and lead detection.

The FC-100 features a powerful built-in cooling fan that ensures cool operation. It dramatically reduces running temperature, makes the lamp more comfortable to hold, and increases the bulb life. The lamp head weighs only 1.5 kg (3.3 lb). It has an impact-resistant thermoplastic housing, a crack-resistant filter and a unique Bulb-Saver bulb protector. A special design allows easy bulb and filter replacement without tools.

The lamp produces a steady-state UV intensity of 4,800 μW/cm². Its visible light emission is under 21.5 lux (2 ftc). (Source: *Materials Evaluation*, October 1996)

Complete computer database radiation safety tracking system

NDT First is a complete computer database radiation safety tracking system developed by Diamond H Testing Laboratories Pocatello, ID. There are two modules to choose from.

NDT First Module I is a complete record-keeping system for the NDT industry. There are more than 35 important reports and forms produced in the 12 files which comprise the innovative, comprehensive database tracking system, which meets all the NRC requirements for radiation safety record keeping. These include: technician time-keeping for payroll, NDT experience tracking in each method; customer job tracking for job costing, invoicing and individual project tracking. The radiation safety tracking system, as described under Module II, is also included in this module.

NDT First Module II is the radiation safety tracking system. There are more than 25 important reports and forms produced in the eight files comprising the tracking system, which meet all the NRC requirements for radiation safety record keeping. These include: NRC Form 4, "Occupational External Radiation Exposure History"; NRC Form 5, "Current Occupational External Radiation Exposure"; monthly dosimeter summary; equipment and radiographer tracking for calibrations, leak tests, field inspections, audits, etc., on a six-month tracking log; calibration records; source and camera records; and QA programme "Certificates of Compliance" tracking. System requirements: Windows or Macintosh. (Source: *Materials Evaluation*, October 1996)

NDT today

Formerly regarded as a black art, non-destructive testing (NDT) is now an essential attribute of cost-effective design, development, manufacturing and process operations. The term is self-explanatory. NDT detects and evaluates flaws in materials, components and structures without impairing their quality and future use. There are widespread applications in industries ranging from aerospace, automobiles, energy and chemicals to food processing and electronics. NDT is routinely applied in environments that range from factories to under the sea.

Testing and analysis carried out during product and process development ensure that design requirements are met for a specified life. But this objective may be compromised if there are flaws in the raw materials or if manufacturing or operation introduce unwanted defects. NDT, however, can ensure that materials are of adequate quality and that flaws introduced in manufacturing operation do not degrade product performance or lifetime. As such NDT is far from exotic. It is part of a family of quality assurance techniques that also involve materials testing, destructive examination and sample testing.

When used to check equipment that is already in service, NDT ensures that operational conditions are not causing unacceptable deterioration and that equipment can continue to operate safely. Regulations may require periodic inspection, and flaws detected by in-service NDT are evaluated against fitness-for-purpose criteria. These are not necessarily the same as the standards set by design requirements or those applied when the purchaser accepts the product. The most harmful service flaws are corrosion, pitting and planar defects such as cracks. Analytical methods like fracture mechanics are used to evaluate these, distinguishing harmful flaws from those which may safely be left without repair or removal. As such, in-service NDT is complementary to condition monitoring, which detects the mechanical, chemical or electrical effects of wear and breakdown while machinery or plant is running.

NDT techniques are chosen to detect flaws as efficiently and cost-effectively as possible. Some are well-established, but new ones are continually being developed to solve new problems and improve performance. The choice of technique must take into account the nature of the material, the shape of the component and the conditions under which the testing must be carried out.

It is equally important to consider the nature and characteristics of the flaws which could exist. Surface flaws can often be detected by visual inspection, which is the simplest and oldest form of NDT. Sensitivity is enhanced by the use of magnifying glasses or microscopes and the range of applications may be extended by using introscopes, fibre optics and cameras. But many flaws can only be detected if their visibility is enhanced. There are several means of doing this. Penetrant testing, for instance, uses coloured or fluorescent dyes, which enter open flaws, rendering them visible against a contrast medium sprayed onto the surface. In contrast, magnetic particle inspection can only be applied to magnetic materials. A suspension of coloured or fluorescent particles reveals the disturbance of magnetic flux around surface-breaking flaws in the material. Under suitable conditions, however, these simple methods of NDT have great sensitivity.

A more quantitative technique for detecting flaws at or near the surface is eddy current testing, which involves moving a probe over the surface of an electrically conducting material. Eddy currents are induced and the changes in the impedance of a sensing coil can be measured by a sensitive instrument. Special probes are available to detect flaws in locations such as rivet holes. Eddy current testing is particularly useful on non-magnetic materials, especially aluminium alloys, and is used extensively in the aircraft industry.

Flaws which do not break the surface, however, must be detected by volumetric inspection, carried out by radiography or ultrasonics. X-ray generators and radioactive isotopes provide the necessary penetrating radiations.

Radiography is a sensitive method of finding quality defects in metals, ceramics, composites and many other materials. It is particularly useful in testing welds and other forms of joint. However, its ability to detect service-induced cracking is questionable. Fortunately, ultrasonic testing can meet this need. A probe placed on the surface launches a short pulse of ultrasonic energy into the material and echoes from surfaces. Flaws are detected after at time delay which depends on the distance of the reflector from the probe. Ultrasonic signals are displayed on a screen for interpretation by trained operators or can be electronically processed to provide automatic rejection of flawed products.

Ultrasonics is routinely applied to steel products, metallic castings, forgings and fabrications. It can be applied to all solids, including ceramics. In its simplest form, ultrasonics can be used to measure thickness without requiring access to both surfaces. In other applications, complex components are scanned from several different directions and a composite picture is built up with surfaces and flaws displayed in their correct relative positions. Digital processing technology greatly extends the capability of ultrasonic testing, especially when it is automated.

However a weakness of ultrasonic testing is the difficulty in accurately measuring flaws. The Time of Flight

Diffraction Technique (TOFD) overcomes this by advanced signal processing. The technique is now available for rapid, routine use under site conditions.

Elsewhere, thermography is increasingly employed. Imaging the temperature of visible surfaces reveals flaws that affect the flow of heat inside the material. It can reveal faults in electronic circuit boards, but is equally useful on large structures such as boilers, where it can detect defects in insulating lagging, which give rise to expensive heat losses.

NDT applications fall into three broad categories: manual, automated systems, and special systems. Of the three, manual inspection is most cost-effective when circumstances permit. Successful application of manual NDT, however, requires highly experienced operators, since there is usually no independent check on an operator's work. Certification of competence therefore becomes an important issue. In Europe, independent certification schemes such as PCN (Personnel Certification for Non-Destructive Testing), which comply with European Standards, hold sway. In some industries and other parts of the world, companies certify their own staff, following codes of practice set by bodies such as the American Society of Non-Destructive Testing (ASNT).

Surface techniques of NDT (visual inspection, penetrant testing and magnetic particle testing) are nearly always carried out manually. Manual ultrasonics and eddy current testing are also common.

Automated NDT systems, however, are found on many production lines. They incorporate materials handling equipment and reject defective materials or components without human intervention. Such systems use a variety of NDT techniques and evaluate the results against pre-determined settings. Computer control greatly extends their flexibility and sophisticated NDT can be fully integrated into many production lines.

Special testing systems are necessary when manual inspection and automatic systems are inappropriate. These are used in some of the most demanding applications. Typically, these systems employ an automated device which scans sensors over the component, signal drivers, data processing, recording and display devices. Interpretation may be carried out either on- or off-line. Major users include the nuclear power, offshore and petrochemical industries.

Two factors are driving NDT towards improved technical performance. Firstly, existing materials are being used at higher levels of performance and their tolerance to flaws is correspondingly reduced. Secondly, new materials, which are being developed continuously, have their own characteristic flaws and NDT is called upon to detect them in ceramics, carbon fibre composites, glass-reinforced plastics, stainless steels and duplex materials.

The ultimate goal of NDT is to devise a system which tests the whole of a component or structure from a single point—and preferably without requiring contact with it. Acoustic emission and X-ray tomography come closest to this ideal. Unfortunately, neither has yet achieved its full capability.

Acoustic emission exploits the small pulses of acoustic energy which are generated when a material is under stress. These may result from dislocation movement in front of a moving crack, damage to the fibres or bonding of a composite material, or a variety of other causes. A transducer detects these "distress signals", which are amplified and processed. Unfortunately, however, there is no guarantee that all serious flaws will be detected. Nor are all materials which give the distress signals necessarily defective. Nonetheless, acoustic emission remains an active research field and there are several successful applications, including leak detection and the monitoring of pressure vessel proof testing.,

Conventional radiography provides a two-dimensional image of three-dimensional objects, but tomography has been developed to provide cross-sections. Existing systems require large, heavy equipment and exposure times are long. If the present rate of progress is maintained, there is every prospect that techniques such as this will become practicable or even routine in the future. (Source: *Engineering*, November 1996)

Central certification of NDT personnel

Central certification and the ISO document—what do they mean? ISO 9712, non-destructive testing qualification and certification of personnel, is an International Standards Organization (ISO) document that establishes a system of central certification of NDT personnel in each country that chooses to operate it. The document encompasses three tasks:

- The setting up of an Independent Certifying Body (ICB) to administer NDT personnel certification;
- Independent third-party examinations of NDT personnel, covering, at levels 1, 2 and 3, the general theory, sector specifics and the practical application of an NDT method;
- The standard calls on the ICB to authorize the training of NDT personnel, prior to examination.

There are few, if any, countries or documents which mandate or call on central certification of NDT personnel to this standard.

In the United States of America there is no machinery yet in place to set up or approve an ICB. The American Society for Non-destructuve Testing (ASNT) has not adopted this standard, but agreed to align itself with ISO 9712. In Europe, ISO 9712 has been redrawn as European Standard EN473 (general principles for qualification and certification of NDT personnel).

Several European countries set up ICBs and many of these have been in turn accredited to European Standard EN45013 (general criteria for certification bodies operating certification of personnel) and their programmes for NDT personnel accredited to both EN473 and ISO 9712.

The UK is in an enviable position of leading the way in central certification of NDT personnel. The Certification Scheme for Weldment Inspection of Personnel (CSWIP) was set up by The Welding Institute (TWI) in the late 1960s. Although the scheme did not meet any external standard, it came to be accepted as the standard for training and certification of NDT personnel, mainly in the offshore, construction and service inspection industries throughout the 1970s and 1980s.

By the late 1970s, CSWIP was becoming outdated. It was too sector-specific, being based on the examination of plain carbon steel welds, and materials and processes were changing. Central certification was called up for operators examining castings and forgings, many in support of offshore and power-generation industries. Sector-specific schemes were starting to emerge—for example, AP12PRX for complex weld geometries for the offshore industry and the CEGB Cmv endorsement for materials with high Cr, Mo and V. The use of CSWIP-approved operators had become the norm across the general NDT industry. CSWIP-approved operators were being used to examine castings and forgings, many unaware of the methods of manufacture and type of defect that can appear.

By the end of the 1970s it was evident in the UK that there was a need to establish a new national scheme for certification of NDT personnel, offering examinations covering all sectors of industry. The British National Committee for NDT (BNC for NDT) and the British Institute for NDT (BInstNDT) invited British industry to participate, and in 1981 a central certification board (CCB), representative of major users and specifiers of NDT, was formed.

The PCN (personnel certification for non-destructive testing) scheme continued throughout the early 1980s. Although the scheme was gaining popularity, it was not making money, and by 1986 it became apparent that this scheme could no longer be run by a committee of volunteers.

The BInstNDT scheme for accrediting NDT training schools was adopted to standardize the training of personnel.

By 1989, industry confidence in the PCN scheme improved and the CEGB agreed to desist from their industry-specific programme of certification of NDT personnel and enter a transitional agreement with PCN. The Ministry of Defence (MOD) agreed to a transitional agreement for its AQD scheme, and was in turn joined by the Civil Aviation Authority (CAA).

The CCB decided in 1991 that all PCN certificates should be issued by one agency. TWI and PCN submitted proposals and CCB decided, on the grounds of economy and ideals, to place the central issuing facility with the BInstNDT at Northampton.

The year 1992 saw the birth of the ISO 9712 standard and from that, in 1993, came the European Standard EN473. PCN quickly reissued its documentation in line with this European standard, and in 1994 an extension to their scope of accreditation was granted, showing the PCN scheme to be in compliance with ISO 9712.

New ultrasonic flaw detection

NKK has developed a new on-line ultrasonic flaw detection system that uses chirp wave pulse compression technology to dramatically enhance the signal-to-noise (SN) ratio in ultrasonic flaw detection, thereby improving the ability to detect flaws in steel products. NKK has already tested the system on existing ultrasonic flaw detection equipment for plates, seamless pipes and rails, and has confirmed the system's commercial advantages.

The system uses a multiple-cycle frequency modulated chip wave, which sounds like the chirp of a bird, in place of the single pulse wave traditionally employed as transmission signals. This signal processing method calculates the correlation between the received echo signal and the transmitted signal and compresses the pulse width. Use of long-pulse width transmission signals enables the transmission power to be increased and the correlative processing of the received signals reduces noise, increasing the SN ratio by 3-10 times with just one exposure to the transmitted signals, when compared with conventional systems.

To date, chirp wave pulse compression technology has been used in radar applications, but NKK is the first to apply it to commercial on-line ultrasonic flaw detection, made possible as a result of the company's development of high-speed digital filtering and optimized correlative processing technologies.

The system is regarded as a highly promising, next-generation technology that contributes to better quality assurance, greater testing reliability and lower manpower costs because it eliminates the need for off-line detection. NKK plans to transfer the technology to ultrasonic testing machine makers and other users in Japan and overseas.

For more information, contact: NKK Corporation. 1-1-2 Marunouchi, Chiyoda-ku, Tokyo 100, Japan. Fax: (03) 3214 8400. (Source: *Steel Times*, January 1997)

Analyser handles tough FEA tasks

Most finite-element packages are good at finding stress and deflections in solid materials such as steel or aluminium. But when it comes to infrequently encountered materials and physical conditions, users need another tool. PDEase2 could be a candidate.

The package provides a low-cost solution to problems involving a wide variety of physical phenomena, from heat transfer to chemical reactions, either in steady-state or time-dependent conditions. The software needs no external graphical interfaces of finite-element (FE) solvers, letting users create 2-D FE models with one or more planes of symmetry, such as those encountered in extrusions. Users must first write a differential equation or two to describe the behaviour of the material, but these can be quite simple.

Analysing a production problem involving a ceramic component shows a few of the software's features. Manufacturers use injection moulding to produce ceramic components whenever high yields and tight tolerances are needed. Ceramic powders are mixed with binders, usually a mix of waxes and polymers at 50 to 60 per cent by volume, to make the ceramics injectable. The binders, however, must be removed by heat treatments. Temperatures above the binder melting point extracts them. Binders in a liquid state lose the strength needed to support the ceramic extrusion, so it slumps or deforms. Experimental measurements describe time-dependent deformations (creep) during heat treatment. The measurements characterize creep behaviour and can be used to optimize processing conditions to minimize total deformation.

Results of tests demonstrated the effectiveness of a simple and versatile FE code in modelling and predicting creep behaviour of relatively complex architectures. These models can be used later to test different types of setter geometries that minimize total deformation. The software allows plotting flow direction, flow rate and stresses. PDEase2 now runs in Windows 95 and NT.

For more information, contact: Macsyma Inc., 20 Academy St., Arlington, MA 02174-6463. http://www.macsyma.com. (Source: *Machine Design*, 6 February 1997)

The colour of stress

Engineers typically use strain gauges to measure the stresses on automobile parts, chain bridges, bridges and other weight-bearing objects. But the load stress on this heavy-duty hook was visualized with an infrared camera and processor developed by Stress Photonics, a company in Madison, WI. The infrared camera works by picking up the minuscule temperature changes (down to less than four-thousandths of a degree) that result when a metal is stressed. Stretching cools the metal, just as a gas cools when it expands; compression heats it. The hook, used for lifting cargo, bore loads of up to 700 pounds. (Source: *Discover*, March 1997)

STEP for materials

STEP is an unofficial acronym (Standard for the Exchange of Product Model Data) for an emerging ISO (International Organization for Standardization) standard (formally, ISO 10202) for the computer-to-computer exchange of technical information. STEP is intended to facilitate enterprise integration by simplifying computer systems integration no matter what types of computers or software are used or what natural languages (tongues) are spoken by people in the organizations involved in the enterprise. Its development is being driven mainly by the world's aerospace, automotive, electronics, building construction and ship-building industries, for which rapid formation of relatively short-lived and flexible partnerships and supplier arrangements around the globe is a way of life (perhaps even a matter of survival). For the materials testing world though, the need for STEP may not be as obvious.

STEP is an outgrowth of IGES (Initial Graphics Exchange Specification), developed in the late 1970s to provide means whereby a given computer-aided design (CAD) system can communicate with another CAD or computer-aided manufacturing (CAM) system regardless of what computers or software the systems are using. It does this by providing a standard for the CAD/CAM files to use strictly in the exchange from one system to another.

Fundamentally, the standard is for exchange of *unequivocal* concepts taken bitewise and in a relatively well-confined context. STEP does this by providing internationally agreed-upon (consensus) meaning and structure to the data when exchanged among computer systems. The closest analogy to STEP is probably the EDI (Electronic Data Interchange) standard for business information that has made worldwide ATM (Automated Teller Machine) access and pay-at-the-pump possible.

Taking a simple example, "today's date" is a well understood concept (called an "entity" in STEP parlance). It usually has three defining parts (called "attributes" by STEP) for which the meanings are fairly well recognized: a number or string of words representing the year in the Christian era (e.g., "1997", or "nineteen ninety-seven"), a word or number representing the month of the year on the Gregorian calendar (e.g., "April", or "04"), and a word or number representing the day in the month on the Gregorian calendar (e.g., "Tenth", "10th", or "10").

The structure is the sequence in which those attributes are arranged during the computerized exchange and the relationship that the attributes have to one another and the entity. In the USA the structure of a date is usually month-day-year; in Europe it is usually day-month-year; and in the Orient it is usually year-month-day, which is the convention adopted by STEP. They all agree as to what "today's date" is because their computers can exchange the date through the STEP format as 1997/04/10, that is, in an unequivocal and unambiguous form.

The Integrated Generic Resource Models, Parts 41-49, provide common structures for the data, whereas the Application Protocols (APs) populate those structures with explicit, agreed-upon meanings for some purpose (called an "application context" in STEP). The APs are where the technical experts (called "domain experts" in STEP parlance) in a given field such as materials testing have to come to an agreement on the terminology to be used. The resulting AP is a standard itself that is basically a set of rules ("protocol" in STEP-ese), which stipulates that when you want to exchange data for such-and-such a purpose via STEP, you must sequence the data in a certain way and use the designated terminology (thus the term "application protocol"). The Standard Data Access Interface feature provides ways whereby a STEP file can be converted into the formats used by other software if not already in the STEP format. These features are the concerns of the computer programmers (called "information specialists" by STEP). If the programmers and the technical experts have done their jobs correctly, the users should not even be aware that the exchanges are via STEP.

Given the dizzying pace of the world of computers, perhaps it is useful to also say what STEP is *not*. It is not a standard such as ASCII for the way in which bits and bytes are arranged to represent letters and symbols. STEP is not a standard such as SGML (Standard Graphic Markup Language) or HTML (Hypertext Markup Language) for the way in which letters, words and pictures are displayed on a computer screen or page. STEP is not a standard such as SQL (Structured Query Language) for querying the contents of databases. STEP is not a standard such as CAMPUS (Computer-Aided Materials Pre-Selection by Uniform Standards) for how data from the testing of polymers are presented on a page. STEP is not even a standard meant to deliver information for humans to read directly (just as anyone who has inadvertently listened to facsimile transmission "squeal" knows that it is not meant for them to hear). To be sure, it is meant to benefit humans, but to minimize their involvement in the transfer of the concepts as much as possible.

The most pragmatic reason for the materials testing community to be concerned about STEP is that most of the major players of the large world industries mentioned in the introduction are. More than 25 countries have STEP development activities under way, and the major OEMs (original equipment manufacturers) of the world's automotive and aerospace industries have issued joint public announcements to their suppliers that STEP will be used in computerized communications with them at some point in the future. True, those announcements mainly focused on CAD and CAM, but it seems clear that if STEP looks like a success for CAD and CAM, it will probably move quickly to other aspects of manufactured products such as materials testing information.

So, why get involved now? Why not just wait? Well, abstention from the development process of materials-related STEP standards puts the materials-testing community at the mercy of the perspectives of the OEMs. The potential benefits are in reductions of time and transaction costs, increases in flexibility, and possible avoidances of competitive disadvantages from being too far down on the learning curve should STEP become the norm for communication. While these benefits have been quantified (more or less) for the world's CAD and CAM communities, they have not yet been quantified for the materials communities. One segment, that of polymers testing, has recognized this and has asked NIST to try and organize an effort to test STEP for materials testing. It is hoped that this and possibly other prototype efforts will begin to provide such quantification. (Source: *ASTM Standardization News*, April 1997)

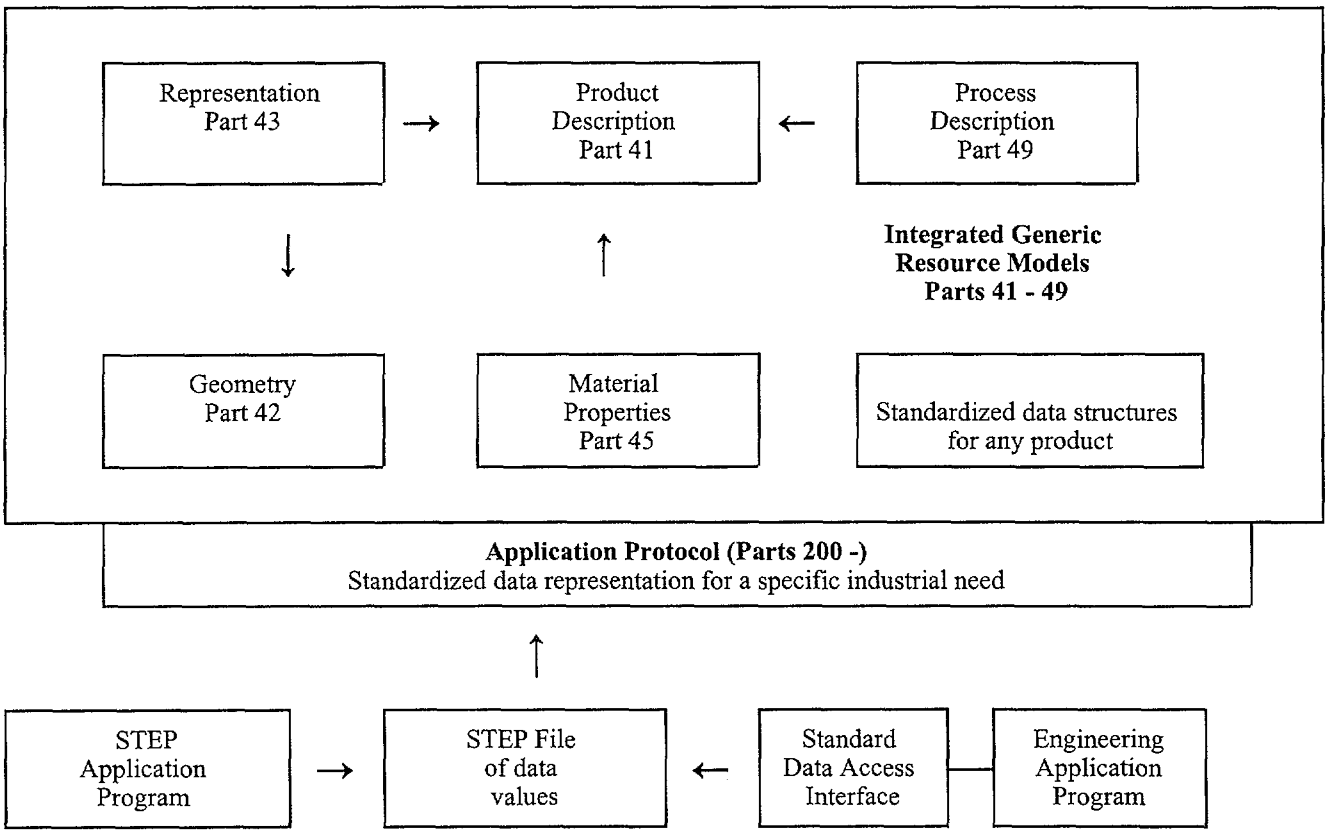
IMIX-PTS EDS system

Princeton Gamma-Tech, Inc. (PGT), Princeton, NJ, offers a wide range of advanced application software modules to solve problems and furnish tools for comprehensive microanalysis. Included are SpotLight Analysis and Advanced Feature Analysis with chemical classification. Each of these modules is available only on PGT's IMIX-PTS with position tagged spectrometry.

Position tagged spectrometry is a new method for microanalysis data acquisition. While rapidly scanning the sample and acquiring a high-resolution electron image, it processes X-ray counts and encodes them with the specimen

The Structure of STEP

The following diagramme illustrates the main features of STEP. The Application Protocols (Aps, middle) provide specific, standardized *meanings* of data being exchanged from some industrial purpose (e.g., exchange of data from materials testing) while the Integrated Generic Resource Models (top) provide standardized data *structures* common to all Aps. The Standard Data Access Interface (SDAI, bottom-right) feature provides ways whereby a STEP file can be converted into the formats of other software (far bottom-right) that are not already STEP compliant (e.g., bottom-left).



X, Y coordinate information. The operator gets a high-resolution digital image, a full EDS spectrum from the image area, multiple spectra from any regions selected within the image, and quick X-ray maps for all elements in the periodic table. The maps and spectra may be displayed live during collection or may be requested after collection from stored position tagged data. (Source: *Materials Evaluation*, April 1997)

Industrex AA 400 film

Eastman Kodak Company, Rochester, NY, has announced the first product in a family of improved industrial X-ray films based on Kodak's patented tabular grain (T-Grain) emulsion technology. Kodak Industrex AA 400 film provides equal or improved image quality, speed and contrast compared to Kodak Industrex AA and AX films used for non-destructive testing applications. Kodak T-Grain emulsion uses flat, tabular grains to capture more direct X-rays and photoelectrons providing high imaging efficiency with more uniformity. T-Grain technology also allows the design of easier handling from more robust films with increased flexibility in a range of processing cycles.

Kodak Industrex AA 400 film provides the following benefits: excellent consistency; long, stable shelf life; stable contrast throughout the processing cycle; and improved physical characteristics, such as resistance to humidity, less high-temperature sensitivity at exposure, resistance to abrasions and kink marks with a reduction of runback, and pi line in automatic processing. (Source: *Materials Evaluation*, April 1997)

TH 9590 and TH 49401 linear X-ray detectors

Thomson Tubes Electroniques, Moirans-St. Egreve, France, has two linear X-ray detectors. The TH 9590 is brand new and designed for high-speed production lines (up to 20 items per second). Designed around a 512 photodiode linear array with 0.45 mm (0.017 in.) spacing, the TH 9590 detector provides a sensing length of 230 mm (9.05 in.). It delivers an 8 bit output signal. Minimum integration time is 0.3 ms per line, so the operating speed reaches 1.5 m/s (4.9 ft/s) without image distortion.

The TH 49401 is an ultra-compact X-ray image intensifier designed for high-resolution radiological imaging applications. Useful entrance field diameter is 40 mm (1.6 in.) for an overall length of 38 mm (1.5 in.), and resolution is 10 lp/mm with no geometrical distortion. Options include an ultra high-speed phosphor, which enables imaging at up to one million frames per second, and a beryllium input window for low-energy X-rays. The 49401 may be supplied optically connected to a CCD camera providing a standard video output. The overall length of this image head is 1 mm (0.04 in.). The TH 49401 is insensitive to high magnetic fields and well suited to harsh industrial environments. (Source: *Materials Evaluation*, April 1997)

Primary beam monochromator

Philips Analytical X-Ray, Almelo, The Netherlands, has introduced the Alpha 1, a primary beam monochromator for its X'Pert line of X-ray diffractometers. Addition of the Alpha 1 allows performance of advanced analysis (such as line profile and Rietveld analyses) with improved accuracy. This is achieved by removing the Kα2 component of the Kα radiation from copper X-ray tubes and by improved axial divergence of the X-ray beams. As a result, the Alpha 1 features a better definition of peak profiles, better peak/background ratio, and a well-defined background. The factory aligned monochromator is equipped with a symmetrically cut Johanssen type Ge (111) crystal. The user can adjust the omega rotation of the monochromator crystal for optimization, for example after exchanging X-ray tubes (Source: *Materials Evaluation*, April 1997)

Mini-NDT

The AFP Mini-NDT processor from AFP Imaging, Elmsford, NY, is a full-feature, compact industrial X-ray processor. The unit features a 356 mm (14 in.) feed width, has the same features found in higher priced models, and can be used on a table or on its sturdy base stand (supplied with all processors). The top front exit maximizes use of space. Because of its cold water operation and "no plumbing" conversion kit, the Mini-NDT can go anywhere from darkroom to job site in both mobile and stationary installations. Roller racks and a film transport system in the energy efficient infrared dryer are specifically designed for processing industrial X-ray films. The worm drive greatly reduces load on the system and allows for easy rack removal for maintenance as well as extending the motor life. Easy access allows the user to carry out maintenance rapidly and easily. (Source: *Materials Evaluation*, April 1997)

X-MET 920 XRT Autosampler probe

The 920 XRT Autosampler from Metorex (Bend, OR), with 16 position removable sample tray, is designed to meet the latest market requirements for a high-performance, automated X-ray fluorescence elemental analysis system. The system's technology is based on the same proven design principles as Metorex's X-MET series radioisotope excited XRF analysers. The 920 XRT Autosampler includes the added benefits of automated sample changing and X-ray tube excitation. The 16 position sample tray is easily removed for convenient sample loading. The special low-power X-ray tube requires no cooling and emits radiation only when powered on. Its compact size makes it one of the world's smallest tube excited XRT analysers. The systems are equipped with matched detector/target combinations. (Source: *Materials Evaluation*, April 1997)

NDT-X4 high-capacity industrial film processor

The AFP NDT-X4 industrial film processor from AFP Imaging, Elmsford, NY, is a marriage of two AFP processors. It combines the proven and reliable transport system of the 14X family with the system design of the Mini-Medical film processor. The NDT-X4 provides a speed of 235 mm (9.25 in.) per minute, and a 7 min. 40 sec. processing cycle capable of processing as many as 45 sheets of 355 x 430 mm (14 x 17 in.) films per hour.

The rack design, identical to the one used in the 14X family, uses the "orbital side plate" method to transport film through the rollers which are constructed of a state-of-the-art cast-moulded polyurethane material. It features 430 mm (17 in.) wide rollers so that 355 x 430 mm (14 x 17 in.) film can be run sideways or two 200 x 250 mm (8 x 10 in.) films can be processed side by side. The NDT-X4 can be installed in several configurations: through-the-wall with the processor in the daylight; through-the-wall with the processor in the darkroom and film exiting outside the darkroom; and free-standing with the entire processor in the darkroom. (Source: *Materials Evaluation*, April 1997)

Test sample is precise part

A test specimen is the most important component of tensile testing, for it determines the actual physical properties of the material being tested. The specimen must conform to exacting physical dimensions and must be free of induced cold working or heat distortion. Qualified laboratory personnel and accurately calibrated tensile machines are important, but the ultimate test results are based on the quality and accuracy of the test specimen.

Preparation of test specimens with a conventional miller creates heavy chip loads due to slow cutter speed, and can induce severe internal distortion to the machined edges. Blanking, shearing or die cutting also cause deformation of the critical specimen edges; thus, both methods often produce erratic and inaccurate test results.

The ideal tensile specimen should duplicate the pure basic metal or plastic being tested with a total absence of tool marks, incipient cracks, induced stresses, internal deformation or heat distortion.

Tensilkut achieves these ideal requirements and produces almost perfect test specimens.

The equipment that Tensilkut Engineering manufactures prepares physical test specimens for tensile, compression, flexure, impact, tensile-impact, fatigue and creep tests in accordance with ASTM, DIN, BS, JIS and other standards. The machines were designed for use by laboratory personnel without machining experience. CNC controlled units are also available.

For more information contact: Tensilkut Engineering, 1901 Clydesdale, Maryville, TN 37801. Fax: 423/982-6347. (Source: *Advanced Materials & Processes*, June 1997)

Silicon's complex surface structure

Silicon is one of the most common elements on Earth, yet the structure of its surface is probably the most complicated of all—a three-layered geometric construction of atoms with tiny holes at the peaks. Researchers at Northwestern University, Evanston, IL, and NEC Corp. in Japan have made the clearest images to date of this complex surface.

According to a Northwestern press release, the scientists used a high-resolution electron microscope (HREM) to develop images of the Si (111) surface with 7 x 7 re-construction that revealed a beautiful symmetry when the jagged surface was seen from above.

These images are the first to show all three levels of atoms at the same time. They are the clearest and most detailed images to date of the surface of silicon.

The images also confirm the model for the surface structure of silicon first proposed by K. Takayanagi and associates in 1985. This so-called dimer-adatom-stacking (DAS) model consists of 12 adatoms (first layer); a stacking fault bilayer (second and third layer), within which nine dimers (third layer) border the triangular subunits as the core structure of 1/6[112] screw dislocations; and a deep vacancy at each apex of the unit cell. The model was developed using transmission electron diffraction (TED) data with an assist from the observation of adatoms by scanning tunnelling microscopy (STM).

While STM, which uses low-energy electrons, successfully image the adatoms on the surface, many key features, such as the dimers in the third layer, were too deep in the structure.

However, HREM, which uses high-energy electrons, can obtain atomic-scale information about surfaces by direct imaging.

The Northwestern/NEC researchers used plan-view imaging (electron beam perpendicular to the surface) coupled with noise-reduction filters (which enable images to be obtained at very high resolutions) and numerical inversion (to separate the top and bottom surfaces). The images clearly show both the adatoms and the buried dimers.

There are only a handful of HREMs currently in use. One is at Northwestern and a second is at NEC's Fundamental Research Laboratories in Tsukuba, Japan.

For more information, contact: Erman Bengu, research assistant, Dept. of Materials Science and Engineering, Northwestern University, 2225 N. Campus Drive, Evanston, IL 60208-3018. Fax: 847/491-7820; E-mail: bengu@apollo. numis.nwu.edu. (Source: *Advanced Materials & Processes*, June 1997)

Easy-to-use current-to-voltage interface for characterization of material

Dielectric spectroscopy is a versatile and straightforward analysis method to gain insight into the molecular dynamics of many materials. The basis for this technology is that application of an electric field to a dielectric tends to orient the permanent dipoles inherent in most molecular constituents of matter.

The Mestec Dielectric Module DM 1360, designed by the Max-Planck-Institute in Mainz, Germany, addresses laboratories equipped with a gain-phase analyser, such as the Solartron SI-1260 or lock-in amplifiers such as the Stanford SR-850. These instruments, however, offer insufficient current sensitivity in order to exploit the low frequency range (10^{-3} Hz to 10 Hz) of the built-in generators for dielectric relaxation measurements, and their dynamic range cannot cover 10 decades of dynamic signal behaviour.

When combined with Solartron's 1260 gain-phase analyser, the DM 1360 can measure e* (w) in the wide frequency range of 10^{-3} to 107 Hz, with a resolution better than tan (d) 10^{-3} for the bulk of this range.

The DM 1360 can be used for the following key applications:

- Impedance analysis of electrical components, networks and materials;
- Precise dielectric relaxation-spectroscopy e* (w) on liquids, solid state materials, polymers, ceramics etc.;
- Measurements of absolute values for dielectric constants and dc-conductivities, including contact free;
- Material characterization by AC and DC losses.

In contrast to similar commercially available dielectric instruments, the DM 1360 needs no feedback selection and is therefore an extremely compact and easy-to-use measurement tool. (Source: *Chemical Engineering World*, Vol. XXXII, No. 6, June 1997)

MRI and AFM combined in analysis of microstructure

A technique that is said to have the potential to analyse the interior of materials in slices as thin as one atom has been developed by a team of scientists from Los Alamos National Laboratory, Los Alamos, NM, and the California Institute of Technology, Pasadena.

Designated magnetic resonance force microscopy, the method combines magnetic resonance imaging (MRI) and atomic force microscopy (AFM).

Magnetic resonance provides information about the electronic structure and magnetic spin dynamics of a sample, but limited sensitivity restricts resolution. On the other hand, AFM has very high sensitivity, allowing the determination of surface structure with atomic-scale resolution.

Magnetic resonance force microscopy is a mechanical technique in which a very small diameter cantilever (similar to that for AFM) helps to reveal magnetic properties of a surface. The cantilever is bent slightly by the weak magnetic force generated by the electronic spin of atoms in the sample. Radio-frequency waves excite spins at targeted locations inside the material, which makes the cantilever sensitive to the subsurface locations. Magnetic resonance force microscopy currently matches the sensitivity of state-of-the-art MRI. In addition, atomic-scale resolution is possible if single-spin sensitivity is achieved.

The technique is well suited to study the interfaces of multilayered, thin film materials for electronic applications, such as disk readers in computers.

The capability of non-destructive analysing regularities at the interface between layers of magnetic material sandwiched with non-magnetic metal layers (copper and gold, for example) and relating them to local magnetism and electrical properties can help to improve information storage density on magnetic disks.

For more information, contact: Chris Hammel, LANL, MS K764, Los Alamos, NM 87545. (Source: *Advanced Materials & Processes*, June 1997)

Serial sectioning links with computer-aided reconstruction

Combining serial sectioning and computer-aided reconstruction, scientists at the Naval Research Laboratory, Washington, DC, and the University of Virginia, Charlottesville, have reportedly developed an experimental technique that provides a three-dimensional (3-D) analysis of a microstructure.

Serial sectioning consists of incrementally polishing through a thin (0.2 mm) layer of material, chemically etching the polished surface, applying reference marks and examining selected areas by optical or scanning electron microscopy.

Computer-aided registry aligns the micrographs from each section, and the images are viewed both as video sequences that step through the material slice by slice, and as 3-D reconstructions built up by advanced computer visualization techniques.

The 3-D reconstruction technique allows a more detailed understanding of microstructural development, which in turn allows closer control of both mechanical and physical properties. (Source: *Advanced Materials & Processes*, June 1997)

KT develops fibre line monitoring system

Korea Telecom (KT) has developed an optical fibre line operation and monitoring system (FLOMS) which allows remote testing of fibre cables and automatic cable fault locating.

The system, to be deployed soon, will enable KT to continuously monitor its fibre cable facilities, thereby preventing communication disasters caused by the degradation of the facilities.

Currently, optical fibre lines in operation cannot be tested. Performed on reserve lines only, testing is carried out manually by repair experts once a year.

The developed system will be used for monitoring the carrier's trunk lines laid between cities. For subscriber fibre lines, it is working on another system. (Source: *Korea Newsreview*, 19 July 1997)

Automatic materials preparation

For over half a century Buehler has been a leading manufacturer of scientific instruments and supplies for cross-sectional analysis. Buehler products are used throughout the world by metallurgical laboratories, quality control departments, and failure analysis facilities for the analysis of all types of materials, including ceramics, composites, semiconductors, metals, rocks and minerals, and plastics.

The all new VANGUARD™ system brings affordability to the area of fully automatic sample preparation systems. At a reasonable price, laboratories can completely automate their preparation process. Productivity increases as well, because laboratory personnel are freed to perform more critical duties, such as sample analysis.

The VANGUARD is a fully automatic system that will grind, polish, clean and dry samples without any operator intervention. This PC-based system allows the user to select from an array of stored polishing routines that can be tailored to the material being prepared. Single sample pressure allows one to six samples to be prepared simultaneously.

The PHOENIX 4000, a new standard for semi-automatic grinding and polishing offers an extremely vibration-resistant, stable and corrosion-proof platform. This system offers single sample pressure, which will allow the operator to inspect individual samples at any time during the preparation process. Available in single- or dual-wheel models, with 10 or 12 in. (25 or 30 cm) diameter wheels, the PHOENIX 4000 sets the industry standard for semi-automatic preparation.

Buehler OMNIMET® image analysis systems provide the speed and productivity laboratory personnel demand in today's competitive environment. These Windows-based systems represent the culmination of Buehler's long commitment to automating the analysis process.

For more information, contact: Buehler Ltd., 41 Waukegan Road, PO Box 1, Lake Bluff, IL 60044-1699. Fax: 847/295-7929. (Source: *Advanced Materials & Processes*, June 1997)

Spectro Analytical Instruments

Spectro Analytical Instruments was founded in 1979 to create atomic emission spectrometers using new technology. For the first 19 years the company experienced exceptional growth and today Spectro is the largest supplier of emission spectrometers in the world.

The initial product, Spectrotest, a mobile spectrometer, used fibre-optics to allow on-site testing of metal chemistry. It was rugged, reliable, easy to use, and met an industry need to sort scrap metals by alloy type. Capabilities were soon expanded to include incoming positive metal identification, to avoid in-process mixes, and for outgoing QA/QC verification of metal products. Spectrotest and related products such as Spectroport continue to be a growth area for the company due to continued expansion of capabilities. As examples, after many man-years of R&D the ability to test for carbon was added a few years ago, and the ability to test for phosphorous and sulphur were recently added.

In the early 1980s, the technologies developed to make rugged and reliable mobile spectrometers were applied to laboratory systems, the Spectrolab. For the first time, spectrometers with the accuracy to certify metal chemistry could be used in non-laboratory locations. In a somewhat startling discovery at the time, it was found that fibre optics, when used to capture the light from sample excitation, actually improved spectrometer accuracy by allowing most alloys of a given base metal to be measured on one set of calibration curves.

Fibre optics and advanced manufacturing techniques allow Spectrolab to be made with multiple optical systems, essentially multiple spectrometers within one chassis. This unique design avoids the compromises necessary with single optical systems; one optical system can be high dispersion and resolution, while another can have wide wavelength coverage.

Spectro scientists and engineers recently patented UV Plus, a maintenance-free, non-vacuum optical system with the ability to measure wavelengths down to 120 nm. UV Plus opens new windows in spectrochemical analysis by allowing the precise measurement of nitrogen in steel and titanium alloys, and oxygen and hydrogen, critical elements in high-purity copper.

Spectro innovations were first applied to inductively coupled plasma (ICP) almost 15 years ago. A key advancement in this field was the development of a direct-wavelength-drive monochromator system. Monochromators provide the ultimate in analytical flexibility by measuring any wavelength under computer control. Unfortunately, existing monochromators did this by rotating the grating and using a "peak search" routine. Because there is no peak at the detection limit, peak search monochromators have degraded detection limits and can find the wrong peak at low concentrations. Using advanced technology, Spectro developed a monchromator with a fixed grating and moving detector. This monochromator is fast, requiring less than a second to change between any two wavelengths, and because peak search is not required, it produces detection limits comparable to polychromator (simultaneous) systems.

The Spectromass is an inductively coupled plasma mass spectrometer with exceptional sensitivity for the analysis of liquids and gases. The ICP delivers ions to a precision quadrupole mass spectrometer. The quadrupole functions as an atomic mass filter, allowing ions with a specific atomic mass to pass through to a detector. This technology allows elemental analysis at parts-per-trillion levels and isotope ratio determinations.

For more information, contact: Spectro Analytical Instruments, 160 Authority Drive, Fitchburg, MA 01420. Fax: 508/342-8695. (Source: *Corporate Spotlight*, June 1997)

LECO Corp.

LECO Corporation has been pioneering new and innovative analytical technology for more than 60 years. Starting with the first rapid carbon determinator for the iron and steel industry, the product line now includes a variety of analytical instruments, spectrometers, metallographic and optical products and high-quality consumables. LECO is an ISO-9001 certified manufacturer.

LECO's comprehensive metallographic line includes a new Spectrum System 2000™ automated grinder/polisher. This versatile system can be upgraded, beginning with the modular base unit to which the fully automated grinder/polisher head and then another base unit can be added. The system interface provides memory for up to 10 detailed methods for consistent sample preparation.

The Microsoft® Windows® '95 software makes the IA-3001 one of the simplest-to-use image analysis systems available today. With the visual report designer, polished reports including histograms, bar charts, *x-y* plots and control charts are quick and easy to prepare.

LECO's GDS-750A glow discharge spectrometer is capable of highly-accurate bulk analysis as well as quantitative depth profile analysis on a wide variety of matrices, including difficult sample types not practical by other OES techniques. An optional RF source permits analysis of non-conductive materials.

For more information, contact: LECO Corporation, 3000 Lakeview Ave., St. Joseph, MI 49085-2396. Fax: 616/982-8977; Web site: http://www.leco.com. Leco is a registered trademark of LECO Corporation. (Source: *Advanced Materials & Processes*, June 1997)

RJ Lee Instruments Ltd.

RJ Lee Instruments is a "different kind of SEM manufacturer". Providing sophisticated yet user-friendly analytical instruments to application-specific markets, its novel approach and ability to solve complex and varied materials analysis problems is the basis for its success.

As an applications driven company, RJ Lee Instruments is committed to providing the exact tool required to maximize productivity relative to given materials analysis requirements. Utilizing an in-house staff of applications engineers, software engineers and customer support engineers, the company provides ongoing consultation as applications evolve or change.

An affordable, easy-to-operate scanning electron microscope, the PERSONAL SEM is as "at home" on the shop floor as it is in the laboratory or classroom. Equipped with a disposable filament and column liner tube, it requires no special utilities and is easily maintained by the operator. Ease-of-operation enables in-house staff to perform routine analyses which may currently be contracted to outside laboratories.

The PERSONAL SEM is configured to suit specific application needs:

- **Automated Feature Analysis** for particle size analysis with associated chemical composition.
- **Automated Inclusion Analysis** for rating cleanliness in metals and alloys.
- **Gunshot Residue Analysis** for automated analysis of residue collected from hands, face or clothing for forensic application.
- **Natural Fibre Analysis** for differentiation of fibres on the basis of morphology.
- **Optical Preview and Relocation (P&R) Station** for comparison of optical and SEM micrographs from exactly the same sample area.
- **Variable Pressure Analysis** for analysing samples in their natural state, without coating or other tedious preparation.

For more information, contact: RJ Lee Instruments Ltd., 515 Pleasant Valley Road, Trafford, PA 15085. Fax: 412/744-0506, or on the Internet at http://rjleeinst.com. (Source: *Advanced Materials & Processes*, June 1997)

Laser detection

The Accusort Model 22 barcode scanners from Visolux now enable automatic identification procedures to be used by manufacturers of light-sensitive materials.

New and existing installations can be equipped with a laser which operates at the high end of the infrared scale with a wavelength of 1,300 nanometres. This means the scanner can operate in the presence of light-sensitive materials without either exposing them or changing their chemical composition. (Source: *Engineering*, July/August 1997)

Composites assessed

Many designers are still wary of using composite materials despite their high strength-to-weight ratio and ease of customization to specific applications because of the diversity of factors that need to be considered when assessing their possible use. But this task could now be made much simpler with the aid of a new software package for assessing composite material properties in the context of specific structural applications.

The CoDA software developed by National Physical Laboratory (UK) (NPL) is claimed to make it possible for design engineers to instantly see the effect on the deflections of a panel or beam of changing any relevant material data such as fibre or matrix properties or laminate lay-up. It can be used for materials formed by all process routes from hand lay-up to injection moulding. As such it can help in design development and assessment, modification of stress loading patterns and development of new materials.

The software has four modules. The synthesizer module enables the properties of composite materials to be generated, taking into account the volume faction and type of reinforcement, its orientation and the properties of the matrix polymer. The software computes the thermal and moisture expansion properties, as well as the stiffness and strength properties in the two principal directions. Both continuous and discontinuous reinforcement can be analysed.

Meanwhile the lay-up module allows the synthesized materials to be layered in the required manner, with material type, layer thickness and orientation and stacking sequence specified by the user. The panel and beam modules can then predict the performance of structural elements of either type in specific situations.

Further development plans for the software include modules for assessing bolted fixings and impact modelling. The software, which is supplied and supported on NPL's behalf by Anaglyph, runs in the Windows operating environment. (Source: *Engineering*, July/August 1997)

Making the move to automated testing

The growing importance of statistical process control had led Anderton International to upgrade the intensive hardness-testing operation at their Bingley plant in West Yorkshire, UK.

Production testing is now centred on three new, microprocessor-controlled, digital Rockwell machines, which ensure consistent presentation of test data for SPC analysis. Supplied by Indentec, the machines are all standard, bench mounted models, which operate on an automatic test/load cycle, with visual and audible signals confirming a correct test routine, and warning light to indicate a test failure.

Several batches can be processed simultaneously, at the rate of five specimens an hour per batch, or five per batch if production lasts less than an hour.

For more information, contact: Intendec Hardness Testing Machines Ltd., Lye Valley Industrial Estate, Bromley Street, Lye, Stourbridge, West Midlands, DY9 8HX, UK. (Source: *Steel Times*, July 1997)

On-line metal fatigue monitoring

Developed in conjunction with British Steel Technical for the continuous monitoring of dynamically stressed components, Sunnyside Systems' new mill monitor enables mill operators to predict impending fatigue failure, and monitor wear on major plant equipment.

The rainflow analysis technique employed calculates the long term and assesses the residual life remaining, using linear accumulative damage theory and the strain-life curve for the driveshaft material. A continuously updated graphical readout displays remaining fatigue life.

Continuous monitoring also enables the trending of data relative to condition. For example, the transient shocks seen during the normal production process are related to clearances in the drive system, and as these increase through bearing or coupling wear, limits may be set to raise alarms through the overall maintenance network. The monitor is currently employed at British Steel's Universal Beam Mill on Teeside, and is also applicable to other drives such as cranes or loaders.

For more information, contact: Sunnyside Systems Ltd., Nettlehill Road, Houstoun Industrial Estate, Livingston, West Lothian, United Kingdom, EH54 5DL, UK. Fax: (0) 1506 441040. (Source: *Steel Times*, July 1997)

Predict damage to components before it occurs?

A wide range of chemical and mechanical treatments and coatings is available to toughen metallic surfaces against environmental stress, fatigue or corrosion. These treatments can provide the appropriate qualities at a realistic cost, where it would not make economic sense to toughen the bulk of the component to the same level of quality.

However, if the surface becomes damaged or even eliminated during the service life of the component, then the structure's resistance to degradation will be greatly reduced and failure becomes much more likely. One solution to this problem would be a means of monitoring surface quality, both during the initial treatment and during service.

This is the objective of a research group at the NDT Centre at University College London. The intention is to develop a surface quality monitor (SQM), which can provide an estimate of the remaining service life of a component. By acting as a fatigue sensor or even a stress corrosion sensor, it would reveal the state of the component's protective surface layer and show whether re-treatment or even replacement is necessary.

Surface treatments usually change a material's hardness, chemical composition or residual stress or, alternatively, involve depositing a protective coating over the original material. Several of these key changes in surface properties are known to cause changes in magnetic permeability, so that electrical and magnetic property measurements can open up a window through which these surface properties could be monitored. Earlier work on the use of alternating current field measurements (ACFM) has shown that these measurements can be used to measure the growth of cracks in metallic materials. This opens up the possibility of adapting this technique to measure degradation of protective films or coatings during a component's service life.

Fatigue, for example, is a major cause of in-service failures and is a direct result of alternating stress cycles applied to metal components. It also depends to a lesser extent on the component's static stress level, itself often a combination of externally applied steady loads, and its internal residual stresses. Service load induced stresses can often be predicted on the basis of experience and engineering analysis. But residual stress has been much more difficult to quantify, due to the lack of appropriate non-destructive measurement techniques and the extreme difficulty of prediction based on knowledge of production methods. The net result is that alternating stress can be predicted, but with limited accuracy because of the assumptions which have to be made in the analysis. Equally, steady stress is difficult to predict because of the often unknown nature of the residual stress.

If these stresses could be measured, then the likelihood of fatigue damage could be estimated much more accurately. This creates some intriguing possibilities. At present, non-destructive testing methods can be used for crack detection and sizing, but a workable SQM would reveal surface changes before the onset of cracking, so the component could be treated to restore its original resistance to failure relatively easily.

An instrument of this sort would tell you when you are losing the benefits that you put into the material. If you start losing those benefits, you can reintroduce them before the situation changes from losing benefit to producing damage.

Research has shown that mechanical stress in ferrous materials influences the distribution of the magnetic domains within the material, due to a phenomenon called piezo-magnetism. By using the latest AC field measurement devices to reveal very small changes in the magnetic permeability of the material, it has been possible to detect very small changes in permeability and hence the stresses within the material. In the case of structural steels, for example, it has been possible under laboratory conditions to measure changes in stress of the order of a few per cent of the total zero-to-yield stress range of the material.

The research group hope to be able to use the technology employed in the very latest AC field measurement devices to develop a non-contacting AC Stress Measurement (ACSM) sensor, which could be used to measure the induced magnetic field within a component. So far, ACSM devices have only been used to measure applied stresses, but the intention is to use the sensors for periodic assessments of the residual stress level while a component is in service, to show whether or not the component is close to micro-cracking and whether renewed surface treatment is needed.

The research, which builds on previous work to develop a crack detection system with TSC of Milton Keynes, will concentrate initially on magnetic field measurements in threaded components, such as the drillstrings used in the offshore oil industry.

A complication is that usually only the thread root is treated for fatigue, but the full body of a component may be treated for resistance to environmental attack. So the project will try to predict the relationship between the properties and thickness of the surface layer, the level of residual stresses and the AC magnetic field measurements, so that the magnetic field properties which the sensor needs to measure can be identified. Experimental sensors will then be built to carry out fatigue and environmental loading tests, so that these desired properties can be confirmed or measured and a methodology can be built up for surface quality monitoring. (Source: *Engineering*, July/August 1997)

System analyses chemistry and crystallography

An integrated PC-based system in which the chemistry and crystallography of polycrystalline materials are determined simultaneously has been developed by a partnership of Edax International, Mahwah, NJ, and TexSem Laboratories (TSC) Inc., Provo, Utah. Edax manufactures energy dispersive X-ray analysis systems including two PC-based systems for qualitative and quantitative elemental microanalysis. TSL provides products and services to quantify and analyse the microstructures of crystalline materials, especially orientation imaging microscopy systems.

Through a joint agreement, Edax will integrate the TSL electron backscatter diffraction capabilities into both of their PC-based microanalytical systems. This reduces costs because of the need for only one CPU, keyboard, monitor and mouse to control all the capabilities offered by both manufacturers.

Advanced analysis capabilities are possible with the integrated system. For example, orientation imaging data may be collected simultaneously with EDS spectra or X-ray maps, and advanced crystal identification will be provided.

For more information, contact: Jim Nowak, Edax International 91 McKee Drive, Mahwah, NJ 07430. Fax: 201/529-3156; Internet: http://www.edax.com. (Source: *Advanced Materials & Processes*, August 1997)

Neural-network test devised to detect flaws in MMCs

A neural network-based test method coupled with an imaging source has been developed by Accurate Automation Corp., Chatanooga, TN, to recognize patterns that indicate the presence of cracks and fatigue in fabricated titanium-matrix composites having matrices of alloys such as Timet TiMetal 1100 and Beta 21S. According to the company, the method fills the need for a standard non-destructive test that can ensure composite reliability and also enable wider application of titanium composites in aircraft components such as leading edges, inlets and nozzles.

For more information, contact: Accurate Automation Corp., 7011 Shallowford Rd., Chatanooga, TN 37421. Fax: 423/894-4645; E-mail: marketing@accurate-automation.com. Titanium Metals Corp., 1999 Broadway, Suite 4300, Denver, CO 80202; Internet: htpp://www.timet.com. (Source: *Advanced Materials & Processes*, August 1997)

Laser-based test measures film properties in real time

An ultrafast, laser-generated ultrasound technique in which thin-film thickness and critical properties are measured in real time has reportedly been developed by the Nondestructive Evaluation Branch of the Wright Laboratory Materials Directorate, Wright-Patterson Air Force Base, OH. The method enables films as thin as 10 nm to be accurately measured without contamination or damage.

In the process, a very short pulse of light from a titanium:sapphire laser propagates ultrashort pulses of ultrasound through the film.

The pulses are reflected back to the surface from the substrate below. Film surface displacements caused by the ultrasonic echoes are detected by a pulsed interferometer, and these values become the basis for precise measurements of film thickness. The sensitivity to small displacements is increased by a delay line-based pulsed interferometer, which permits a frequency range four orders of magnitude greater than that for conventional interferometry.

The technique has enabled accurate measurements of films ranging from 25 to 170 nm thick. Because it takes advantage of ultrafast pulses, it can discriminate between coating and substrate properties. The pulse method can also measure the thickness of transparent films, the degree of contamination on the surface of the substrate, the degree of amorphous vs. polycrystalline microstructure and thermal conductivity of the film.

For more information, contact: Materials Directorate Technology Information Center, WL/TIC Bldg. 653, 2977P St., Suite 13, Wright-Patterson AFB, OH 45433-7746 (Ref. Item #97-016); Internet: http://www.wpafb.af.mil. (Source: *Advanced Materials & Processes*, August 1997)

Updated condition monitoring imager

A text file option which can guide the operator through a predetermined route in one or more infrared facility inspections is now available for use with the Thermovision 550 hand-held infrared focal plane array condition monitoring imager from AGEMA Infrared Systems, Secaucus, NJ.

Also now available for the Thermovision 550 are software options that allow the images on the unit's PC card to be displayed directly on a PC or laptop computer with no additional software required, and that permit remote control operation of the camera. All three features are available as standard or options depending on the Thermovision 550 package selected, or can be added later if the user's needs change. (Source: *Materials Evaluation*, September 1997)

Differential blackbodies series

The Smart Blackbody series of differential black bodies from Santa Barbara Infrared (Santa Barbara, CA) offers several advantages in accuracy, stability and ease of use. Their design puts the "brains" of the system, the temperature and calibration electronics, in the black body head instead of the control electronics package. The Smart Blackbody offers lower noise operation, more stable temperature control and improved immunity to electromagnetic interference.

This black body is easier to integrate into your system. Calibration is independent of the controller so the user can swap controllers without recalibrating the black body, and there are no limitations on cable length. The Smart Blackbody is designed with improved temperature sensors, higher resolution temperature measurement electronics and ultrastable calibration. (Source: *Materials Evaluation*, September 1997)

Real-time thermal analysis system

FLIR Systems (Portland, OR) has made available Tracer, a thermal imaging system that can record and analyse long thermal event sequences at real time rates (up to 60 Hz) on a Windows-based personal computer.

Tracer combines a high-resolution thermal imaging camera with a Pentium PC, digital video recording system, and Windows-based analysis software. In a base configuration, Tracer can record more than five minutes of thermal activity at a 60 Hz frame rate with 12 bit pixels, capturing 9.3 Mb of data per second for analysis. The system senses temperature differences of $<0.1°$ C at 30° C ($<0.18°$ F at 86° F), and can measure the temperature of objects from -10 to +2,000° C (14 to 3,600° F) with an accuracy of ±2 percent. (Source: *Materials Evaluation*, September 1997)

Ultrasonic/infrared instrument package

The PdM Pack, a new predictive maintenance package from UE Systems, Elmsford, NY, marks the first time the Ultraprobe 2000 ultrasonic leak detector and the EPD-PM-30 IS infrared thermometer have been combined in one package. The diagnostic instruments are designed to be used in conjunction with each other for surveys that incorporate both ultrasound and infrared in an intrinsically safe capacity. This would include steam audits, bearing testing, electrical inspections, or any other inspection programme that would benefit from a multitechnology approach.

The Ultraprobe 2000 provides testing capabilities ranging from simple leak detection to sophisticated methods of mechanical analysis. The rugged EPD-PM-30 IS features laser sighting that makes it easy to pinpoint small targets at a distance, even in low light conditions. (Source: *Materials Evaluation*, September 1997)

Infrared digital still camera

Inframetrics, North Billerica, MA, has added a new infrared digital still camera, ThermaSNAP, to its line of thermal imaging systems. Ideal for predictive maintenance applications where the object of interest is stationary, ThermaSNAP provides instant crisp thermal images on its built-in 100 mm (4 in.) colour LCD screen.

Hand held, this innovative camera weights just 2.2 kg (5 1b) and operates on a standard camcorder battery. Applications for ThermaSNAP include electrical and mechanical infrared inspections, condition monitoring of refractory linings, insulation effectiveness, roofing studies and building diagnostics.

ThermaSNAP's standard video output is compatible with video recorders and standard television monitors. For remote applications, it has an RS-232 control port for controlling the system from a computer. (Source: *Materials Evaluation*, September 1997)

New imaging technique strong on power, gentle on samples

There is a new type of kinder, gentler microscopy. Near-field scanning optical microscopy (NSOM) is an emerging technique that combines the non-destructive advantages of optical microscopy with nanometer-scale resolution near that of atomic force or electron microscopes.

NSOM works by channelling laser light through a fibre-optic probe, scanning it about 10 nanometers above a sample surface, and then collecting it on the other side. An opening t the tip of the probe is only about 50 nanometers wide, smaller than a wavelength of visible light (which is several hundred nanometres) but large enough for a small portion of the light energy, or photons, to escape.

NIST physicists, working in collaboration with researchers at the University of Virginia and the Naval Research Laboratory, recently created an NSOM image of a "photonic crystal", a test material made by embedding an array of tiny glass cylinders in a matrix glass. To the eye, these two clear glasses are indistinguishable. However, they have slightly different indices of refraction (bending light at slightly different angles). Consequently, the NSOM image shows that, rather than travelling straight through the sample, light is guided through the crystal by the cylinders.

The NSOM technique has been developed and embraced by numerous laboratories throughout the world in order to image and characterize nanometre-scale features on biological membranes, semiconducting devices and substrates, fibre-optic communications components and many other materials. A major goal of the NIST programme is to further refine NSOM measurements and modelling so that NSOM can provide truly quantitative measurements of the optical properties of these structures. For example, accurate measurements of the size of the glass cylinders and their index of refraction should result from NIST's collaborative research on photonic crystals. In addition, other NIST research groups are working on developing applications for NSOM, including nanometer-scale chemical composition analysis.

The image described above will soon be available in the "Gallery" section of the NIST World Wide Web site, <http://www.nist.gov>. For technical information about the NSOM project, contact Lori Goldner, A320 Metrology Bldg., NIST, Gaithersburg, MD. 20899-0001; E-mail: <lori.goldner@nist.gov>. (Source: *NIST Update*, Special Edition, 5 September 1997)

Recycled magnesium alloys have low inclusion content

Recent tests show that the NMI (non-metallic inclusion) content of die castings made of recycled magnesium alloy AZ91D matches that of die castings made of virgin material, according to Dow Chemical Co., Midland, MI. Inclusion contamination must be controlled to prevent degradation of mechanical properties, machinability, corrosion resistance and paint adhesion. Dow reports that NMIs may be effectively removed from the melt by "sparging" with argon gas and passing the melt through a specially designed filter. Metal cleanliness may then be measured using a light-reflectance technique that provides the rapid feedback needed to ensure high quality of the refined product.

An example of a recycled magnesium application is the lock housing cast by Contech Div., SPX Corp., Portage, MI. This recycled alloy withstands loads in excess of the minimum specification requirement of 11 kN (2,500 lbf) in fracture load tests, similar to results for commercial AZ91D castings. Also, recycled and virgin alloy castings have similar resistance to corrosion in ten-day salt-spray tests. These test results could lead to wider acceptance by automobile manufacturers of castings made of recycled magnesium.

For more information, contact: Dow Chemical Co., Materials Engineering Center, P.O. Box 652, Midland, MI 48640. Fax: 517/636-8813. (Source: *Advanced Materials & Processes*, September 1997)

Ultrasonic compression waves probe steel/ adhesive interface

An ultrasonic compression wave technique has been developed by TWI, Abington, UK, and Permabond, UK, to evaluate the quality of two adhesive systems and two surface pretreatments for joining stainless steel. The technique involves a single probe, which transmits and receives ultrasonic compression waves at a specified frequency. The ultrasonic response is characterized by a series of repeat reflections from the steel adherend. The repeat reflections depend on many factors, including the quality of the bonded interface. Therefore, they can be evaluated to detect areas of disbonding. TWI has constructed colour-coded, two-dimensional images of each surface examined, based on such representations.

Ultrasonic results confirmed the superior performance of one of the adhesive systems and demonstrated the necessity of degreasing and shot blasting each steel surface prior to bonding. Environmental tests showed that surface preparation is also important to improve resistance to extreme temperature variations.

For example, the bond quality of the degreased and shot-blasted specimen showed no deterioration, but that of the specimen bonded in the "as-received" condition showed severe disbonding over the entire surface.

For more information, contact: TWI, Abington Hall, Abington, Cambridge CB1 6AL, UK. Fax: +440 (0) 1223 894363; Internet: http://www.twi.co.uk. (Source: *Advanced Materials & Processes*, September 1997)

Testing machines

The Super "L" console/pumping unit arrangement has been totally redesigned.

The principal advantages of this new arrangement are: ergonomic coordination of indicating/operational controls for ease of operation, an extremely quiet hydraulic power package, a simple cost-effective scheme to convert pumping units from manual to servo control capabilities, all contained in a single enclosure with a significantly smaller "footprint".

Five new models of S series benchtop machines are now available for performing tension, compression, flexure, shear and other tests at 5, 10, 25, 50 and 100 kN (1,000, 2,000, 5,000, 10,000 and 20,000 (lbf).

An automated Brinell optical hardness testing system, the Air-O-Brinell DS/AOB, optically measures exact impression diameters of test indentations and displays hardness values and other information.

Most Tinius Olsen testing machines are available with computer-assisted data acquisition and control systems that allow data to be automatically collected, analysed and displayed and provide total closed-loop control of testing operations.

New Microsoft Windows™/Windows'95™ software includes the latest user-friendly features that are associated with Microsoft® releases, plus Tinius Olsen enhancements such as built-in statistical process control programs.

Olsen's new MP 993 Melt Indexer features the new microprocessor-based MP 993 Controller/Timer whose interactive software guides the operator through the test and calculates and displays results. It can be used for automatically timed flow rate or flow rate ratio tests.

A new microprocessor-aided Model 92T Impact Tester can quickly execute a wide variety of pendulum impact tests on plastics ... and digitally display high-resolution results. A Model HD 94 microprocessor-controlled deflection-temperature/Vicat tester automatically determines the deflection (heat distortion) or Vicat softening temperature of up to six plastic specimens at one time.

For more information, contact: Tinius Olsen Testing Machine Co., Inc., P.O. Box 429, Willow Grove, PA 19090-0429. Fax: 215/441-0899; E-mail: info@TiniusOlsen.com; Web site: http://www.TiniusOlsen.com. (Source: *Advanced Materials & Processes*, September 1997)

Software to reduce errors

SATEC Materials Testing Equipment introduced NuVision Mentor. Developed in Windows NT, Mentor is a complete, turnkey hardware control and software package for the operation of creep, stress-rupture and stress-relaxation test frames.

Mentor's ability to automatically apply loads, regulate temperatures and monitor, record and store all test data lessens the opportunity for error and improves traceability.

Mentor automatically calculates test results and generates reports for improved overall quality and increased laboratory productivity. NuVision Mentor may be added to virtually any common test frame, including dead-weight and front-load testers, and those with fixed and variable-ratio Class 1 lever arms or Class 2 arms. It is appropriate for conducting creep, stress-rupture, and stress-relaxation tests on metals, plastics, ceramics and other advanced materials at elevated or ambient temperatures.

NuVision Mentor is the latest offering in SATEC's arsenal of proprietary software products for the materials testing industry. Mentor joins NuVision Partner, the revolutionary software package in Windows95, that performs tensile, compress, flex, bend, shear and a host of other testing applications. GenTest, SATEC's exclusive General Test Package, is now available for NuVision Partner. Designed for the power user, Gen Test is a sophisticated suite of software components that allows laboratory managers, engineers and experienced operators to create complex materials testing procedures without writing a special program.

While development of leading-edge proprietary software is a priority for SATEC, the company regards software control as its latest strategy for serving its customers in the highly competitive global market. SATEC's principal product lines include hydraulic, servo-hydraulic and electro-mechanical universal test frames; fastener, mechanical fatigue, impact, drop weight and hardness testers; automated robotic test systems; creep and stress-rupture testers; NuVision software packages for use with manual or PC-based programmable controllers; and an extensive line of grips, fixtures, extensometers and accessories. The company also specializes in customized system upgrades and retrofits of virtually any make or model of materials testing equipment. SATEC test systems are appropriate for conducting tests to ASTM or other standards, for quality assurance, on-line production control or research and development.

For more information, contact: SATEC Systems Inc., 900 Liberty St., Grove City, PA 16127-9005. Fax: 412/458-9614. (Source: *Advanced Materials & Processes*, September 1997)

Hardness testing

The newly redesigned bench-top Brinell optical scanning system with a computer interface automatically measures Brinell impressions, reads out results on a monitor and stores or prints test results. Operator influence is reduced or eliminated and testing speed is increased. The apparatus is also available as a portable system.

The portable, full-load Rockwell-style hardness testing system uses the NewAge Industries test surface reference feature and dynamic loading to perform Rockwell testing without the need for access to both sides of the part. Operators can test large forgings and other surfaces that do not allow the use of a clamp style system. Test results may be automatically stored, transmitted in RS-232 or printed.

A low-cost computer-assisted measurement system reads Brinell impressions, Vickers or Knoop impressions, total visual case depth and other metal structures. The micro model retrofits to most microhardness testers and increases both speed and repeatability. For measuring, the operator views a VGA monitor, not an eyepiece.

The DataView™ Windows™-based hardness tester data acquisition software package makes it possible to output directly from any digital hardness tester to a PC, for real time, on-screen data management. It also provides unusual features such as minimum thickness and rounded correction factors. DataView will simultaneously display the current test result, a running history of data, real-time X/bar-R charting in individual scalable windows and print out results in chart or graph formats. Tolerances, time/date stamping and test block verification routines are also included.

Windows is a trademark of Microsoft Corporation. Dataview is a trademark of NewAge Industries.

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C. BUSINESS OPPORTUNITIES

Analytical Applications of Synchrotron Radiation

by Dr. Stephen J. Maginn

Introduction

1997 saw the fiftieth anniversary of the first direct observation of synchrotron radiation (SR)[1] (although it had been indirectly detected and theoretically described earlier).[2,3] It has been exploited for scientific research for over thirty years now.[4,5,6] Those thirty or so years have seen a huge explosion in the use of SR in the developed world, the widespread recognition of its unique properties as a vital asset to both pure and applied research, the awakening of industrial interest in its application for commercial gain, and the construction of over thirty dedicated SR sources internationally. The trend continues, driven by the achievements that have already been made and the promise of future achievements. No one article can hope to cover every benefit that has accrued from the use of SR, or cover or even mention every area of scientific enterprise which has made use of SR—many textbooks have been written in specific technique areas—but this article will attempt to briefly cover areas of analytical applications, and specifically structural characterization using X-rays, illustrating the gains that can be derived from the use of SR over conventional radiation, and stressing the commercial applications of these.

What is synchrotron radiation, and why is it so useful?

On first hearing, it may be thought that "synchrotron radiation" is something that may be regularly experienced by the crews of the Starships "Enterprise". Indeed, astronomers believe the synchrotron mechanism to be responsible for much of the radiation emitted by galactic X-ray sources such as neutron stars and black holes. Synchrotron radiation is, however, fully understood and thoroughly down-to-earth in its nature.

This is not the place for a full mathematical description of the production of synchrotron radiation—that information can be found elsewhere[7,8,9,10]—but a brief, qualitative description follows. When a charged particle is accelerated through a magnetic field, simple electromagnetic theory states that energy must be given off in the form of radiation. This radiation is emitted in a direction perpendicular to both the directions of acceleration and magnetic field—thus when a beam of electrons (or positrons, or ions) is passed through a perpendicular magnetic field, which produces an acceleration in a direction mutually perpendicular to both the field direction and the electron's direction of travel, the acceleration causes the electron's path to deflect and the synchrotron radiation emerges at a tangent to the beam's path. Thus, when an array of curved magnets are put together to form a storage ring, charged particles can circulate continuously at speeds approaching that of light, emitting a continuum of radiation at a tangent from each of the bending magnets (in practice, lifetimes in storage rings are limited by factors such as the quality of vacuum, so refills of charged particles are necessary from time to time).

It is also possible to put so-called insertion devices ("wigglers" and "undulators") in the beam path, to produce variations on the standard synchrotron radiation, perhaps with higher intensity or down to shorter wavelengths; synchrotron radiation consists of a continuum of electromagnetic radiation, from infra-red down to hard X-ray in some cases, the exact profile and intensity being dependent on factors such as the speed and strength of the particle (usually electron) beam, the strength of the magnetic field, and the radius of curvature of the magnet.

X-rays produced by the synchrotron method thus have several advantages over those from conventional sealed X-ray tubes:

(1) They have high intensity. The number of photons per second (the "flux") produced is many orders of magnitude higher than even the most intense laboratory sources ("rotating anode" sources). An additional advantage, related to this, is;

(2) The beam divergence is very small. Synchrotron X-rays are directional and highly parallel. The X-ray beams emerging from bending magnets or insertion devices have a small cross-sectional area which is retained over distances of tens and even hundreds of metres inside evacuated beampipes. This also enables the X-rays to be focused using special optics, packing yet more photons into a smaller area.

(3) The continuum of wavelengths produced enables the most appropriate wavelength for a particular experiment to be chosen, or special experiments which require such a continuum to be performed.

These advantages enable conventional laboratory analytical techniques to be carried out with significant enhancements in quality, and have also led to the development of specialized analytical techniques which cannot be carried out elsewhere. Four techniques will be described in this review (powder X-ray diffraction, single crystal X-ray diffraction, X-ray absorption spectroscopy and non-crystalline diffraction), together with some example applications of each, from both academic and commercial environments. The increasing availability of access to SR will then be described, again from both academic and industrial viewpoints, and finally, there will be a look to the future.

SR sources internationally

Synchrotron radiation sources are not cheap or portable! As described above, large numbers of charged particles need to be produced and guided through strong magnetic fields—so strong that superconducting magnets at very low temperatures are sometimes required—in an evacuated beampath. Storage rings may be metres, tens, or even hundreds of metres across. Thus, SR sources tend to be large national or even international facilities, constructed and maintained by consortia of universities, governmental organizations or consortia of governments. No two facilities worldwide are identical—each is optimized to meet the needs of the organizations which constructed it. Table 1 shows a summary of parameters of a few of the world's facilities:

Source	*Location*	*Energy (GeV)*	*Storage ring diameter (m)*	*Comments*
Advanced Light Source (ALS)	Berkeley, CA, USA	1.5-1.9	190	Optimized for soft X-ray experiments.
Synchrotron Radiation Source (SRS)	Daresbury, UK	2.0	96	Utilized for the full range of SR, especially UV, soft and hard X-ray. The first operational purpose-built SR source.
Elettra	Trieste, Italy	2.0	260	Optimized for soft X-ray radiation.
ESRF	Grenoble, France	6.0	850	Optimized for hard X-rays. Operated by a consortium of European States.
Advanced Photon Source (APS)	Argonne, IL, USA	7.0	1 100	Just commencing operation. Optimized for hard X-rays. Each beamline to be operated by consortia of academics and/or companies.
SpRing 8	Himeji, Japan	8.0	1 440	Just commencing operation.

The world's first such facility purpose-built for research applications is the Synchrotron Radiation Source (SRS) operated by the UK Central Laboratory for the Research Councils at the Daresbury Laboratory (see figure 1), near Warrington in Cheshire, UK. This is the Laboratory at which the author is based, and therefore all of the examples described in this article come from this source, which is in full operation, thriving and expanding after 17 years. At the time of writing, the world's largest operating SR source is the European Synchrotron Radiation Facility in Grenoble, France, operated by a consortium of European States, including the UK—this will very soon be overtaken by Japan's Spring-8 facility. A comprehensive list of existing and planned SR sources worldwide, with links to their homepages, is kept at http://wserv1.dl.ac.uk/SRWORLD.

Although the largest and most productive facilities are in the so-called developed world, emerging countries such as South Korea, Taiwan, India and Brazil are also now in the process of constructing or have recently commissioned sources, as are some of the smaller developed economies such as Switzerland. Japan and the UK even have plans to replace or add to their own ageing facilities with bigger, better, new sources.

X-ray powder diffraction

X-ray powder diffraction is one of the most ubiquitous laboratory techniques for the identification and quantification of crystalline phases. Almost every laboratory that performs analytical work on samples in the solid state either has this technique in-house or has access to it under contract. Each crystalline phase has its own characteristic pattern of diffraction peaks, as measured on a conventional laboratory diffractometer. Most work simply involves the identification of phases present in a pure or mixed sample, and sometimes quantification of those phases, although this is trickier and requires higher quality data than simple identification. This may be required for quality control purposes, to define the products of a reaction, or to identify the content and origin of a material for geological or forensic purposes. Occasionally, more advanced work, such as the determination of texture (i.e. particle or grain orientations with respect to crystal structure—vital in determining and understanding physical properties in structural materials, for example), examination of phase transitions in a dynamic system, or attempted determination of crystalline structure is performed.

In the conventional laboratory, the source of X-rays is the sealed tube source. This provides X-rays of a fixed wavelength, characteristic of the metal of which the tube's target is manufactured. Usually this is copper (1.542Å) or molybdenum (0.711Å). This is perfectly adequate for most purposes—the technique requires monochromatic radiation and the signal from an average sample, either inorganic or organic, in a flat plate sample holder, is more than adequate to identify and quantify the phases present. However, if there are minor amounts of some phases (less than 5 per cent) present, or some important features in the pattern, originating from different phases, happen to fall near to each other, accurate identification and quantification becomes difficult.

For structure determination, single crystal X-ray diffraction is the technique of choice (see later), but it is not always possible, for many reasons, to produce a single crystal of adequate size and quality for this. Powder data may be the best one can do, and the amount of information contained in a powder pattern is limited, certainly far less than is available from single crystal studies. Computational techniques such as Rietveld refinement[11] make the most of this, but need a starting model for the structure, and this is usually not available. The first step in an *ab initio* structure determination is to index the powder pattern. This computational procedure derives the unit cell size and shape for the crystal structure from the peak positions in the powder pattern. Indexing is regarded as a "solved problem", and there are several computational methodologies available for it,[12,13,14] but in order for indexing to work accurately and precisely, it needs the peak positions to be determined accurately and precisely. This is often not straightforward from conventionally collected powder diffraction data, because instrumental peak broadening (partly due to beam divergence) may be too great, or if the compound's symmetry is low and unit cell volume high, as is usually the case with systems of commercial interest such as pharmaceuticals, the diffraction peaks will fall very close to each other and may merge together indistinguishably.

The advantages of synchrotron radiation have been described above. All of those mentioned there, (1), (2) and (3), offer significant enhancements to powder data quality which enable the above problems— and others—to be eased or overcome.

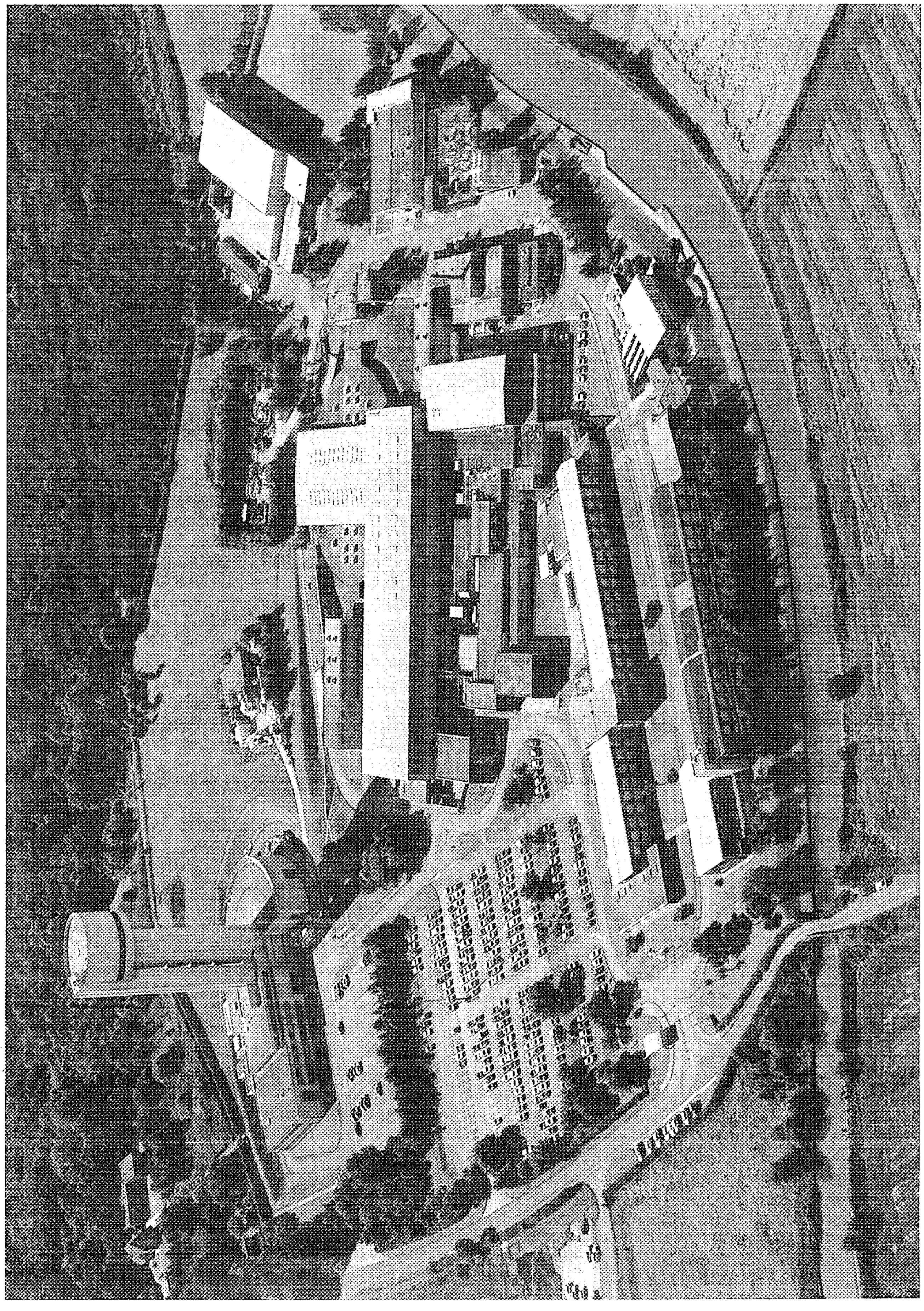

Figure 1: The Daresbury Laboratory. The SRS is housed in the round building, just above the centre of the frame.

The higher intensity afforded by synchrotron radiation enables useful powder data to be collected from samples of much smaller volume. This may be useful if there is only a very small amount of sample available, or for forensic purposes if a relatively non-destructive analysis is needed. Also, it becomes easier to detect, identify and quantify minor phases present, as the better signal-to-noise ratio means that the smaller peaks they produce in the diffraction pattern are more easily discerned and located. Instead of the traditional flat plate sample holder, small volume samples may be confined in a capillary tube, largely overcoming a problem known as preferred orientation, which affects the relative intensities of peaks in a powder pattern.

The low beam divergence at SR sources perhaps offers the greatest gain in quality. This leads to an intrinsic increase in the resolution, and hence precision, of the powder pattern, meaning that procedures such as indexing and Rietveld refinement can be performed more easily and to higher levels of accuracy. A study comparing the "indexability" of powder data collected using the best possible conventional sealed tube source with that collected using the SRS at Daresbury concluded categorically that the SR data gave the more satisfactory solution in every case.[15] Thus powder data collected using SR can offer advantages in the applications of structure solution and refinement, as indexing is easier, and refinement will reveal more subtle structural features, which may be important in explaining observed physical phenomena.

The wavelength tunability of SR is perhaps of less importance here, although an informed choice of wavelength can spread peaks out still further and make them even easier to locate, and any problems due to fluorescence or absorption edges of any elements present in the sample may be easily avoided.

Example: structure determination from powder data

In higher symmetry inorganic systems, where a crystal's asymmetric unit contains relatively few atoms, the amount of information that one can glean from a powder pattern has more chance of providing an insight into atomic positions,[16] but where symmetry is low, as with most organic molecular crystals, chances of success are reduced. One of the first examples of a successful crystal structure determination from powder data for an organic compound of relatively large unit cell volume was achieved using data collected at the SRS.[17] The compound involved was cimetidine, a molecule of pharmaceutical interest, containing 17 non-hydrogen atoms. The structure had in fact already been determined previously by conventional single crystal methods—nevertheless, although *ab initio* indexing was not necessary in this case, it was performed anyway. The SR powder data was processed in order to extract the integrated intensities of each diffraction peak, producing a data set analogous to, but significantly smaller than, the data produced by a single crystal diffractometer. The high resolution of the powder data (figure 2 shows some high resolution experimental data for an inorganic pigment) enabled many more of the diffraction peaks to be accurately integrated than would have been possible with data collected on a conventional source, and the resultant data set proved to be sufficiently large for the structure to be solved.

Refinement was performed by the Rietveld method, and although not all the positions of the 16 hydrogen atoms in the molecule are very satisfactory, the molecular framework and conformation and the packing within the crystal lattice are clear (see figure 3).

Structure determination of organic compounds from powder diffraction is now becoming a topic of considerable interest. Work such as this, and that also performed by groups in the UK, France, Italy and the USA, using both conventionally produced X-rays and SR, and differing computational techniques to produce the starting model (some rely, as above, on integrating intensities from the powder data,[18] whereas others use computational simulations such as the Monte Carlo method,[19] genetic algorithms[20] and others[21,22]), has proved that the most can be made of the minimal information content in powder data. Clearly, an increase in this content, as offered by SR, makes their success more likely. Initially, much impetus for this research came from the dyes and pigments areas of speciality chemical companies, as their compounds tend to be large, flat molecules which are difficult to crystallize in any form other than flat plates, and are awkward for single crystal structure determinations. Their interest continues, but now, much interest is being shown by pharmaceutical companies. Their needs are to characterize their compounds as precisely and tightly as possible, as regulatory bodies demand more and more information and patent challenges over polymorphism issues raise the stakes.

Example: Phase composition of urinary calculus

Synchrotron radiation powder diffraction is a broad church. The example described above is a straightforward extension of laboratory powder diffraction, but there are other applications which have moved further away from traditional laboratory work. One such application is a continuing project to study the phase and texture structure of urinary calculus—bladder and kidney stones.[23]

The mechanism of formation of these stones is by no means fully understood, but their treatment has been simplified in recent years by the use of ultrasonics to shatter the stones *in vivo*. They then leave the body in the normal way. However, some stones stubbornly refuse to break up under ultrasonic attack, in which case removal by surgery is often necessary.

The overall composition of stones is well known, having been established by conventional laboratory powder diffraction. Microscopy studies have shown, however, that stones have a concentric ring structure, and the fine details of the phase quantification and orientational texture of these has been lost in the destructive grinding of whole stones for powder diffraction.

An experiment was thus designed using the SRS, and focused, hard X-rays, of beam size 0.25 x 0.25 mm, on a special materials science goniometer.[24] A sectioned urinary stone of diameter approx. 1 cm (see Figure 4) was placed in the beam, with powder patterns recorded by transmission from each of the concentric rings in its structure. The stone contained relatively large crystallites, leading to poor powder averaging, even over an area as expansive as 0.25 x 0.25 mm, so the powder data was collected using an image plate to account for this. Image plates are two-dimensional area detectors, which capture arcs of, or the entire Debye-Scherrer rings diffracted from a powder sample (see figure 5). Special software can then be used to perform full-ring integration to produce a traditional one-dimensional powder pattern for each image, but free of the poor powder averaging effects (see figure 6). In this case, clear differences can be seen between the images and traces from each ring. Processing has identified that the dominant crystalline phase in the stone's crust is calcium oxalate dihydrate, whilst that inside the stone is calcium oxalate monohydrate.

Image plate technology is by no means restricted to SR sources—it is the ability to pack as many photons as possible

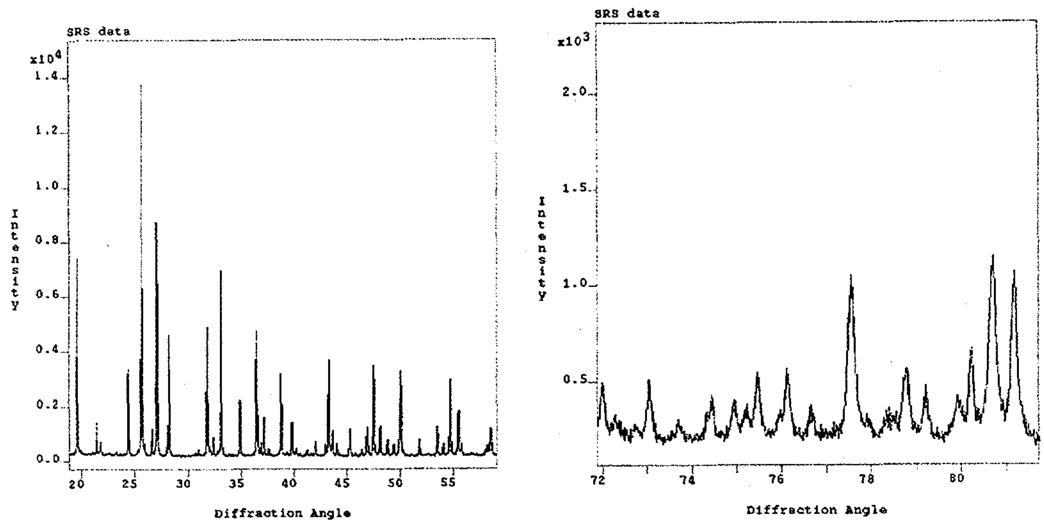

Figure 2: Some typical high resolution synchrotron data, for an inorganic pigment (an Egyptian blue variant), showing the sharp nature of the peaks and the measurable data out to high diffraction angle.

into a small area that is the advantage offered by SR here. Data were collected in just 5 minutes per image for this sample. The X-ray beam size may be slitted down to 50 x 50 microns on the SRS facility used here (station 16.3),[24] making it possible to produce a phase and texture map across a sample such as this urinary stone. This is one possibility for the next stage of this work.

Variants of SR powder diffraction

The special properties of synchrotron radiation make some intriguing variations to standard powder diffraction techniques possible. Here are just two examples:

(1) Energy dispersive diffraction. In a conventionally recorded powder pattern, the wavelength of the incoming radiation is constant and the detector records diffraction at different angles, obeying Bragg's Law. The detector must thus move with respect to the sample to cover a range of diffraction angle, or if position-sensitive, must physically cover that range. SR contains a whole range of X-ray wavelengths, and so with SR impinging on the sample, we can keep the detector fixed and vary the energy of the incoming radiation instead. This is energy dispersive diffraction.

Energy dispersive powder patterns can be recorded much more quickly than conventional, angular dispersive patterns, although some resolution is lost. It therefore becomes possible to perform time-resolved work, watching phase transitions occur as sample conditions (temperature, pressure etc.) are varied. Decomposition or synthetic routes may be followed in the solid state, and the behaviour of materials under extreme conditions may be studied.[25] Two recent industrially relevant examples include a study of gas hydrate formation (see figure 7),[26] and studies of the hydration of cement.[27,28]

In the energy dispersive method, the incoming and diffracted beampaths are fixed, and the detector remains in the same position with respect to the sample, needing to subtend just a very small angle at the sample position. It is therefore possible to enclose a sample much more fully, enabling higher pressures to be reached than inside diamond anvil cells (up to 95 GPa in diamond anvils, up to 400 GPa in tungsten carbide energy dispersive cells).[29] This has proved to be of great importance, amongst others to geological studies, where mineral behaviour under the highest pressures, equivalent to those deep beneath the surfaces of planets, may now be studied.[30]

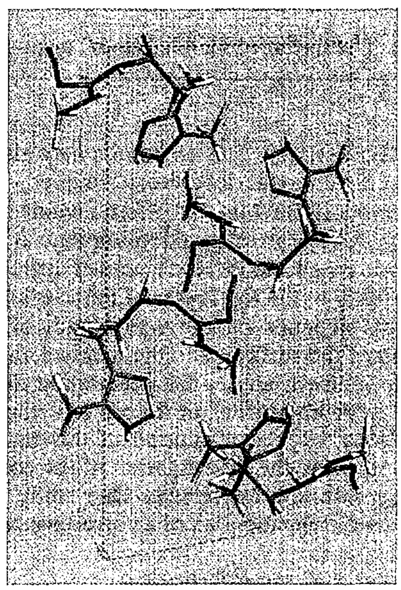

Figure 3: The crystal structure of cimetidine, determined from powder data collected using SR.

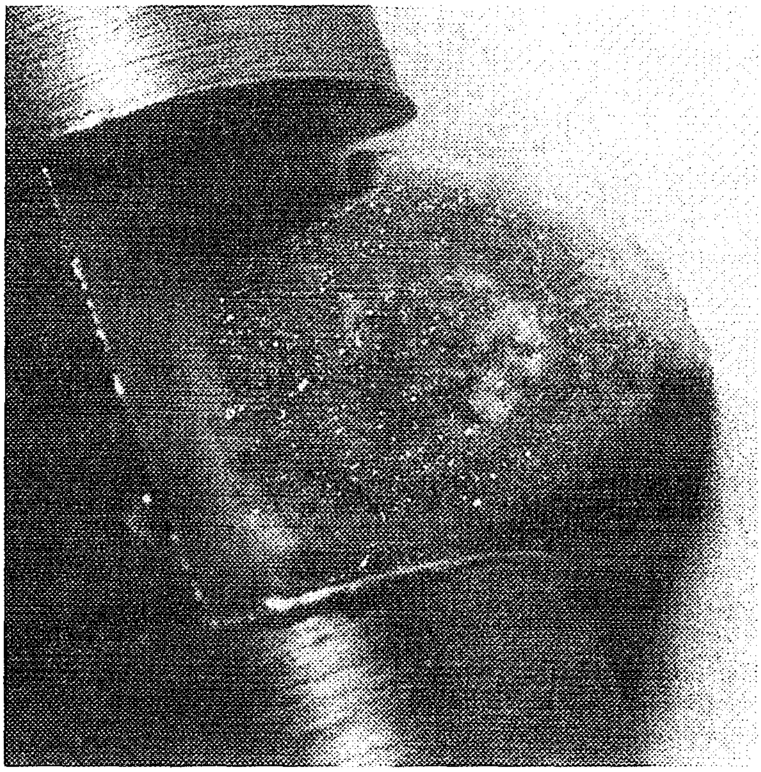

Figure 4: The sectioned bladded stone, mounted and ready for SR analysis.

(2) Reflectivity. This technique may also be carried out using conventional laboratory sources, but as with standard powder diffraction, SR offers a whole new range of possibilities. X-rays are allowed to impinge at a very small incident angle on a surface—the recorded diffraction pattern is then due only to the top layers of that surface. The exact depth of this depends on the wavelength of X-rays, the composition of the surface, and the incident angle. Thus by varying the incident angle, the penetration depth may be varied, and a "depth profile" of the top few microns may be recorded. With SR, the resolution of this may be hugely enhanced, enabling the investigation of the composition of just the topmost few angstroms. One study was able to discern phases in the top 35Å of a corroding sample.[31]

Single crystal X-ray diffraction

As previously stated, this is the technique of choice in the conventional laboratory for the determination of crystal structure, i.e. the arrangements of atoms and molecules within a crystal lattice. Knowledge of this is vital in order to understand the causes of the physical properties and behaviour of compounds in the solid state, and therefore provide a starting point in trying to influence them. Many thousands of new crystal structures are determined each year, as evidenced by the exponential growth of the world repositories for such information, for example the Cambridge Structural Database for organic and organometallic compounds, whose latest release (July 1997) contains 170,000 entries.[32]

Laboratory diffractometers require good quality single crystals of an adequate size for a precise structure determination. That size depends on the nature of the compound concerned, and it is not always possible to produce suitable crystals from the required solvent, of the required polymorph of an adequate size and quality. This may be because the structure is not stable or has some arrangement of molecules that militates against the growth of suitably large, relatively isotropic crystals. A good example is as previously mentioned; large flat molecules tend to produce thin, plate-like crystals.

One possible solution to this is to attempt structure solution from powder diffraction data, as described above, but a recent advance using SR has added another option. By focusing a synchrotron X-ray beam using specially designed X-ray mirrors, a very large number of photons can now be packed into as small an area as possible, and delivered onto as small a sample as possible. Adequate diffraction can now be observed and recorded for very small crystals, down into the microns size range for good scatterers of X-rays, for structure solutions and refinements to be successfully performed for compounds which have defeated the best efforts of the most advanced laboratory sources. A purpose-built facility on station 9.8 of the SRS (see figure 8)[33] is producing its first published results (see figure. 9).[34,35]

Protein crystallography

No review of work with SR can escape without mentioning the huge strides made in the understanding of biological macromolecules, thanks to the application of SR. Many texts[36] and reviews[37] have been written about this, so what follows here is merely the briefest of introductions. The techniques used are developed from the basic methods of structure determination used for smaller molecules, so this passage sits most comfortably at this point in the article.

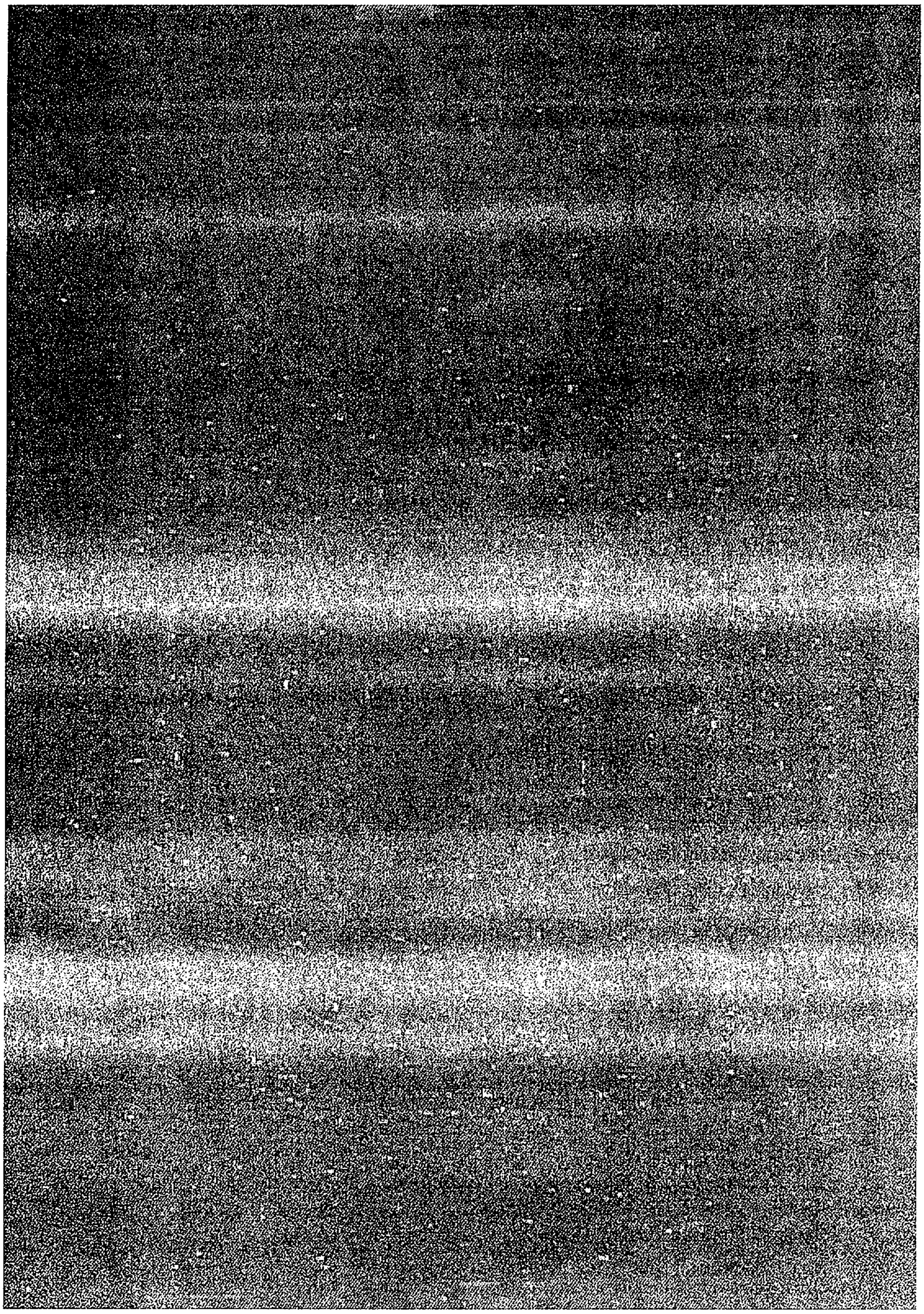

Figure 5: One of the image plate recordings from a ring inside the stone.

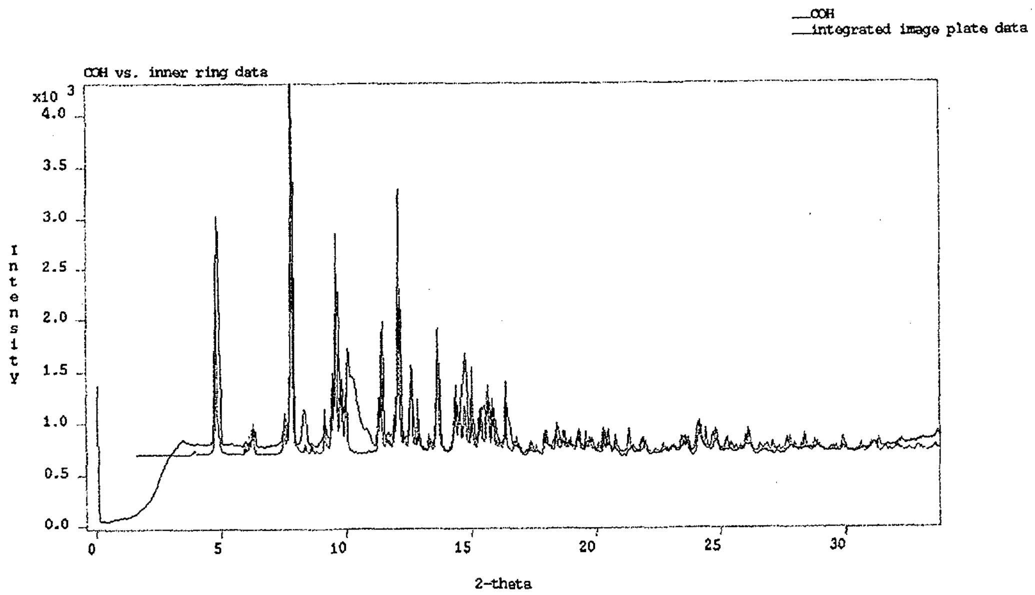

Figure 6: A comparison of the trace derived from the image above (the red line) with the calculated powder pattern for calcium oxalate monohydrate (the green line).

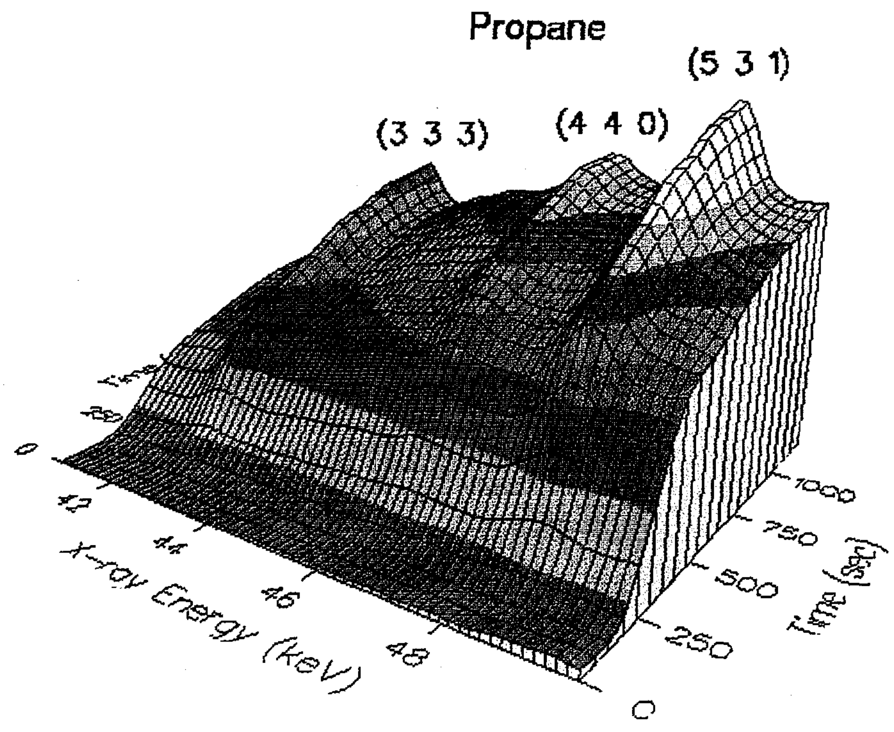

Figure 7: Energy dispersive plot of the growth of propane hydrate.

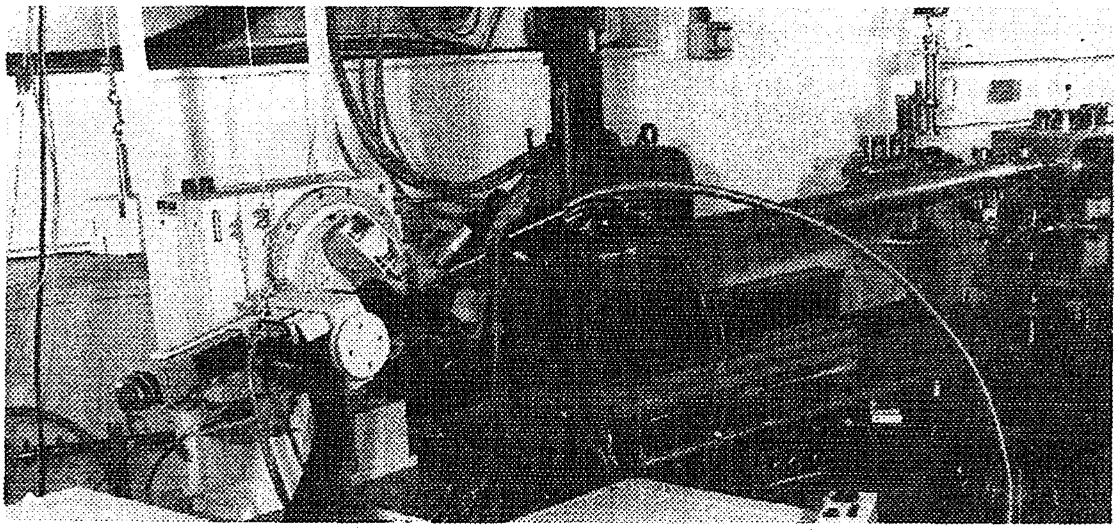

Figure 8: The single-crystal equipment on station 9.8 of the SRS, Daresbury, UK.

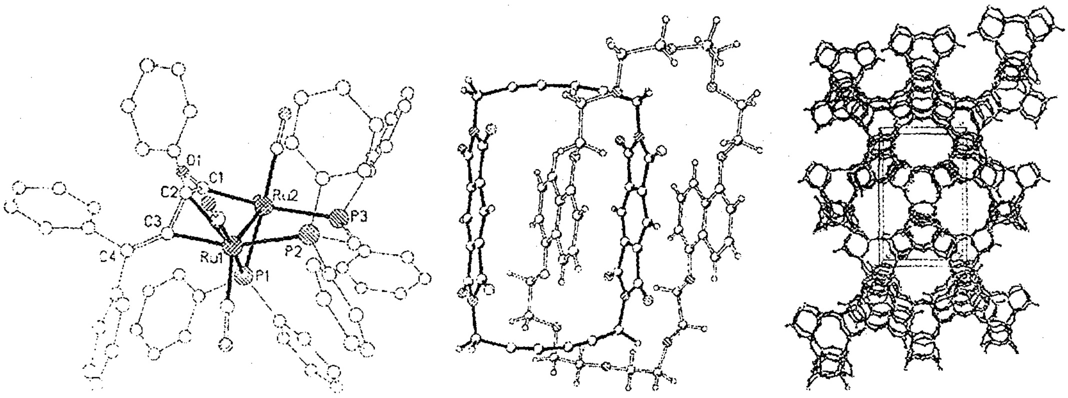

Figure 9: Three recently published structures from station 9.8 at Daresbury [33-35]

Proteins and nucleic acids (RNA, DNA) are vitally important constituents of living systems. Proteins are composed of specific sequences of amino acids, the building blocks of all life on this planet. There are probably over 100,000 distinct proteins in the human body, each of which fulfils an important function. To understand that function, and to find some way of controlling it or preventing threats to it, we need to know the protein's three-dimensional structure.

Protein structure studies are therefore of great significance in medical and pharmaceutical research. They are used as the basis for understanding therapeutic procedures and as a starting point for the rational design of pharmaceutical molecules, a first step in bringing a new drug to market. The best method of achieving a protein structure determination is to obtain crystals of the protein and collect and analyse X-ray diffraction data from them. The human genome project alone is likely to identify thousands of individual proteins, many of which will require X-ray structure analysis to determine their function. But protein crystals are often very small and scatter X-rays weakly, so an intense X-ray beam is needed. On the other hand, the protein molecules within the crystal are usually very large, and a highly parallel X-ray beam is required to accurately resolve the diffraction features. Protein structures can be determined from data collected on conventional, laboratory-based X-ray sources, but to obtain the highest intensities and parallel beam required for the quickest and most accurate studies, synchrotron radiation is required. The 1997 Nobel Prize for Chemistry was awarded to a team investigating how ATP synthase works—ATP, adenosine triphosphate, is the body's energy carrier. Protein crystallographic work in this project, elucidating the synthase structure, was performed using synchrotron radiation.[38]

Figure 10 shows a selection of human proteins whose structures have been recently determined from data collected at the SRS. There is also a huge body of ongoing work on animal, plant, and especially viral proteins, as well as on human systems, targeted at understanding how living systems function, and providing a starting point for therapeutic and pharmaceutical design work.

A typical biological structure determination will require diffraction data to be collected from several different crystals, which have been painstakingly prepared to not live long in the intense SR X-ray beam. The data are usually collected using two-dimensional detectors, and need to be processed using special software which accounts for scaling between crystals, identifies each diffraction spot and eventually produces a list of the many tens or even hundreds of thousands of diffraction intensities required to produce a precise structure. Each of these intensities depends on the positions of the many thousands of atoms within the protein crystal—working back from the intensity list with the help of model structures, advanced computational procedures or a mixture of both, eventually leads to a solution. Studies are often carried out using known protein structures but with other molecules of biological or pharmaceutical significance bound in, so that the sites and mechanisms of binding can be identified. The tunability of SR's wavelength enables multi-wavelength anomalous dispersion ("MAD") experiments to be carried out to pin down the positions of certain atoms within a completely unknown structure—often the first step in a full determination.

Using the ever-greater power of SR sources, which are becoming tailored for biological work as its recognized scientific and commercial importance increases, it is now possible to determine the structures of viruses, with millions of constituent atoms, to atomic resolution. This sort of mind-boggling work is simply not possible without the application of synchrotron radiation.

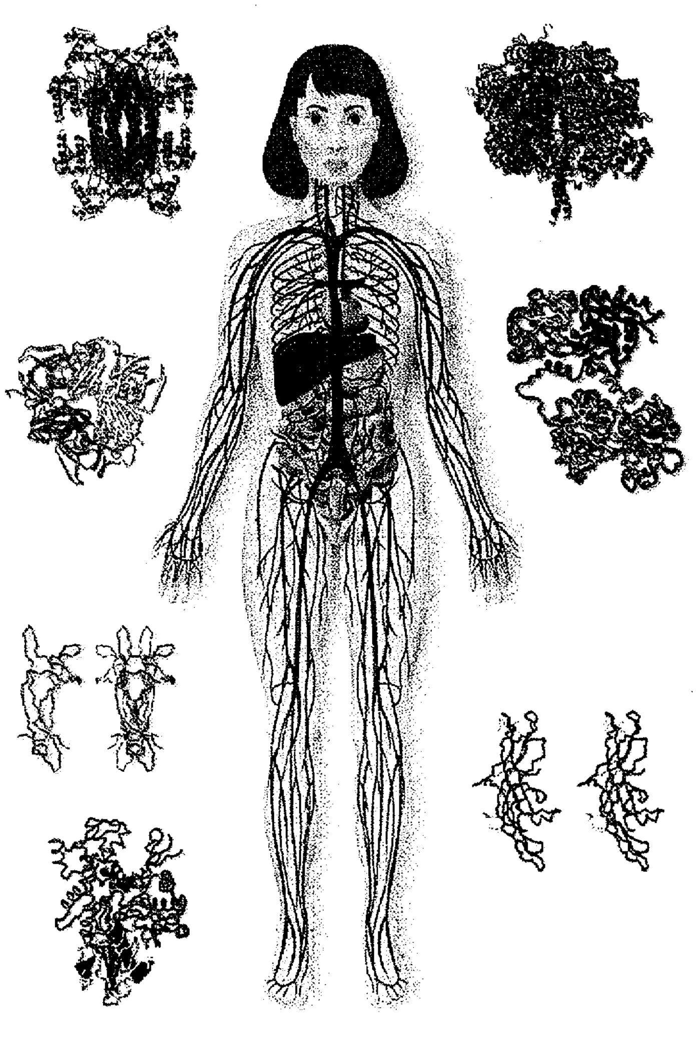

Figure 10: Some recently determined human protein structures. Clockwise from the head, they are FIATPase, transferrin, human chorionic gonadotropin, actin gelsolin, nerve growth factor, ceruloplasmin and delta-crystallin.

X-ray absorption spectroscopies

So far, this review has covered diffraction techniques which are greatly enhanced by the application of SR. X-ray absorption spectroscopy (XAS) is a technique which is barely possible using a conventional sealed tube source (some work has been done utilizing the *Bremsstrahlung* from such sources); it is the wavelength tunability of SR which has led to its dominance in this field and is responsible for the utility of the technique.[39-44]

There are two main branches of XAS-EXAFS (extended X-ray absorption fine structure) and XANES (X-ray absorption near edge structure). Both represent the small oscillations seen in the value of the absorption of a sample when plotted against wavelength (or energy) of incident X-rays on the higher energy side of an absorption edge of one of the elements present (see Figure 11). SR enables edges for any element to be accessed and energy scanning across the edge to be performed, and provides the high intensities required to be able to detect the tiny oscillations for elements of small concentrations against high backgrounds. As the names imply, XANES is concerned with the oscillations within 50 eV of the edge, and EXAFS covers the region beyond that.

The two branches arise from the same phenomenon but provide different information. When an X-ray is absorbed by an element, a photoelectron is emitted. The edge's exact position and the energy of the emitted photoelectron depend on the oxidation state of the absorbing atom, and the way in which the photoelectron is scattered by neighbouring atoms. Thus XANES contains oxidation state information about the absorbing atom, and EXAFS contains information about the chemical and geometrical environment in its vicinity.

EXAFS is particularly useful as a structural probe in systems where diffraction studies cannot help, such as when the atom of interest is present in a crystalline lattice but in very small concentrations as a dopant or impurity, or when the system is not ordered, as in solution, in a glass or in an amorphous polymer matrix. It is not possible to solve structures from EXAFS data, but if a structural model is available, it can be confirmed and refined or discounted. XANES and EXAFS data require a good deal of computational processing and analysis to extract the information contained within it—software for this purpose has been developed exclusively by workers at or closely associated with SR sources.

Example: Dopant environment in a semiconductor

Many electronic materials depend for their function on the presence of defects within the crystalline structure. These may be imperfections or faults within the lattice, or they may be atoms of an impurity element, there by accident or by design. Tiny concentrations of these dopants, in the parts per billion range, can be enough to drastically alter a material's electronic behaviour. In order to understand how this happens, and how to control these effects, it is necessary to determine their exact positioning within the crystal lattice, to tie up with theoretical calculations.

As part of a study into the environment of arsenic dopant atoms in partly amorphous silicon, a model system containing small concentrations of arsenic in crystalline silicon was prepared. EXAFS data were recorded at the arsenic K-edge on station 9.2 of the SRS at Daresbury, and the data were refined against a structural model using the Daresbury Laboratory's EXCURV program.[45] The starting structural model assumed that the arsenic atom simply sat on a silicon site within the lattice, replacing one atom. Refinement confirmed this, and furthermore, revealed the extent of the distortion within the lattice introduced by the presence of the larger arsenic atom in place of the regular silicon. Knowledge of the dopant environment in the crystalline component enabled conclusions to be drawn about its environment in amorphous silicon, and about its preference for one environment over the other, when compared with data for the arsenic edge in the mixed system.[46,47]

Non-crystalline diffraction

The techniques of crystallography depend on there being some large degree of order at an atomic or molecular level within a system. Life, of course, is never that simple, and most processes of interest, be they of academic or industrial use, are by nature dynamic and disordered, or involve a transition between ordered and disordered states. The order may not be at a molecular level but may be on a larger scale, involving domains or particles, extending over tens, hundreds or thousands of angstroms. A range of diffraction techniques have been developed to study systems such as these, and these techniques benefit immensely from the application of SR.[49]

Due to the inverse relationship inherent in the Bragg equation, diffraction from larger scale structures takes place at ever-smaller angles. Thus small-angle X-ray scattering (SAXS), and where appropriate its neutron equivalent, are used to probe systems such as polymer blends, colloids, gels, and heterogeneous phases where electron density contrast can be discerned on scales of tens to hundreds of angstroms. For the same reason that protein crystallography benefits from the parallel nature of SR beams, so does SAXS—subtle features of the diffraction may be detected. The high intensity of SR also means that small volume samples, such as thin films, can be studied successfully.

Dynamic systems also benefit from SR—the speed of data collection for larger volume samples means that time resolution can be greatly improved over conventional lab-based studies. In addition, the development of detectors, which has taken place at SR sites in order to fully exploit the effects which SR makes available and accessible, has been a particular boon here. 2-dimensional area detectors with large numbers of elements in each direction and very short dead-times have enabled the greatest possible amount of information to be gleaned from on-line processes, and phenomena such as polymer crystallization and drawing to be observed *in situ*. The SAXS data can be collected simultaneously with the normal Bragg X-ray region (known as WAXS—wide-angle X-ray scattering), to show in real time how crystalline systems form, break down or change.

Example: Studies of orientation and crystallinity in PET

Polyethylene terephthalate (PET) is one of the most ubiquitous plastics in use. Its applications include fibres, films, tapes and containers, and these each demand different physical properties. These are to a great extent produced during fabrication, which involves cycles of thermal annealing and mechanical drawing, but the link between these processes and the final physical properties is not fully understood. Use of synchrotron radiation has enabled the structural changes which take place during these dynamic processes to be followed. SAXS and WAXS were recorded in real time on a 2 D area detector, with a 40 ms exposure time, as the polymer sample underwent drawing at different rates and temperatures. Observations included the fact that all change in the diffraction pattern—and hence in orientation and crystallinity—was complete within 1 second at the highest draw rates.

SR enabled this study due to its intensity (and hence the time resolution achieved) and the detector technology employed. Scientists from ICI Chemicals and Polymers were involved in this study, showing the interest that industry has in SR work, particularly in this area.[50] Similar studies have been carried out on a wide range of polymers by academics and by industry—a good review of the area is given by Ryan et al.[51]

Example: The active site in a metalloprotein

Metal atoms are vital to the performance of many proteins, providing a catalytic centre at which the protein's functions are carried out. The classic examples of this are iron in haemoglobin, zinc in insulin, and manganese in photosynthetic systems. The techniques of protein crystallography, as described above, have been used to characterize many such examples, but often the important transition states cannot be captured in crystalline form. One such case is nitrite reductase, in which EXAFS has been used to show which of the two copper atoms in the structure performs the catalytic work. The results were used to guide subsequent protein crystallographic work, showing how SR techniques can be complementary.[48]

Access to synchrotron radiation

If this article has performed its function, the benefits of applying SR to a problem should now be clear. So why isn't everyone using it?

The first reason for this has been alluded to already. SR sources are not cheap, portable or easy to come by. They tend to be national or international facilities, run for the

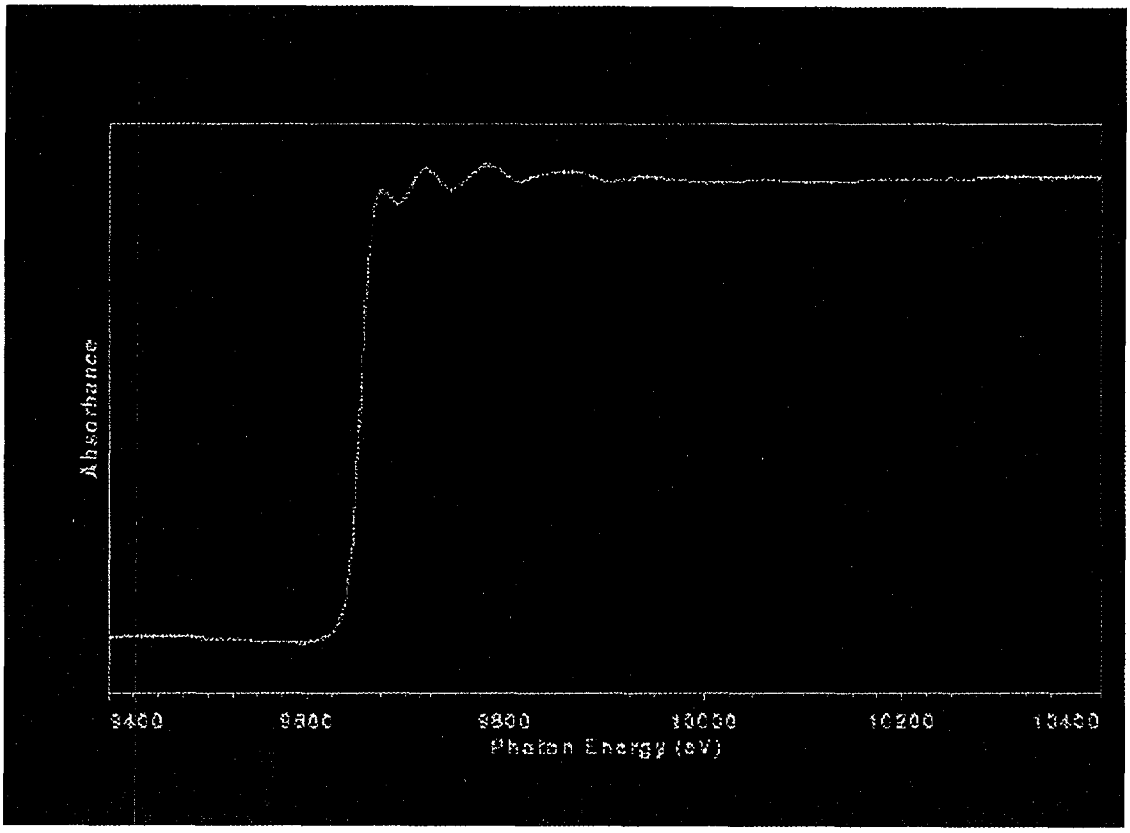

Figure 11: EXAFS fringes at the Zn K-absorption edge

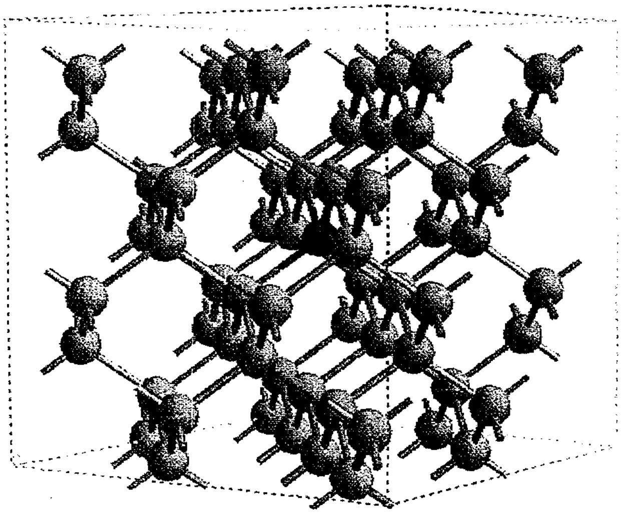

Figure 12: The arsenic atom (in purple) replacing silicon in the crystal lattice

benefit of local or qualified academics and/or industry. In the case of academia, the demand for use of synchrotron facilities invariably outstrips supply. This is certainly the case at the Daresbury SRS, where the UK Research Councils manage academic access through systems of application and peer review. Industry can pay for access to facilities—many SR sites, such as Daresbury or the ESRF in Grenoble, offer paid access to raw beamtime, whereas at others, notably in the USA, some companies have taken part in construction of relevant facilities and benefit through retaining a portion of the available time. In all cases, academic and industrial, support is given to users in terms of infrastructure (equipment and software) maintenance and training, although users design and carry out their own experiments, and collect, process and interpret their own data.

This leads into the second reason why SR use is not yet universal, even in developed countries. Although existing SR use is oversubscribed, it is clear that the volume of potential SR application—the potential "market" for SR—is many times greater still. This market has so far gone largely untapped, partly due to the oversubscribed nature of existing facilities, but mainly due to a lack of knowledge in the wider scientific community of what SR can do for them. With political realities putting ever-greater difficulties in the path of obtaining funding for such major projects as new SR sources, it has become clear that the greater demand there is for SR facilities, the more chance there is that it will be satisfied. Thus attempts are now being made to address this large, untapped market, in both academia and industry.

At the Daresbury SRS in the UK, an industrial SR data collection service, known as "DARTS", has been in operation since early 1997. "DARTS" represents the first serious attempt to market and sell SR use to industry in its

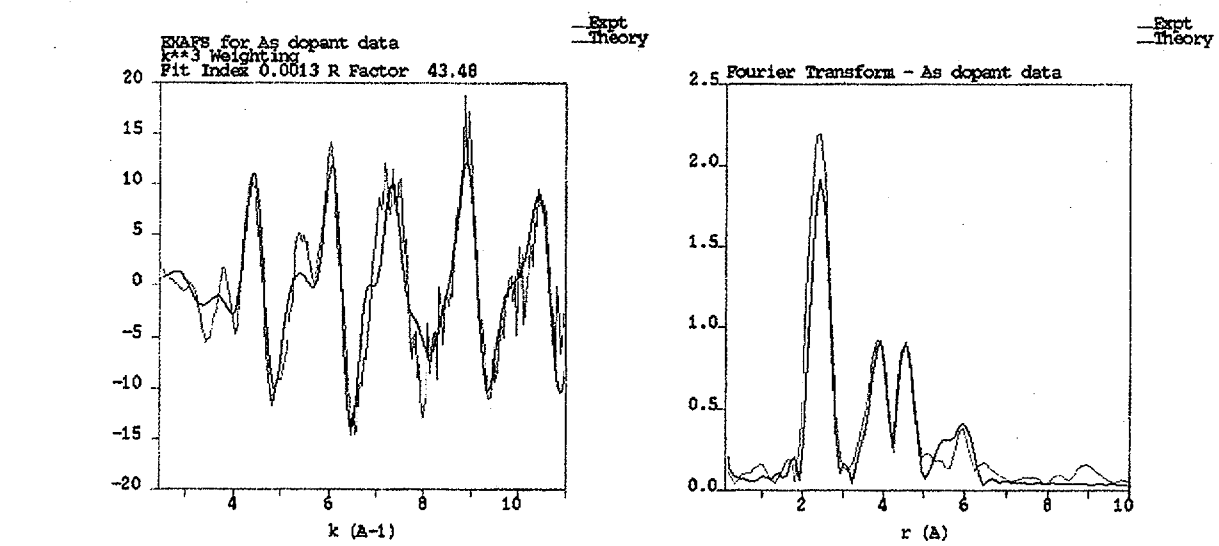

Figure 13: The refined arsenic K-edge EXAFS data

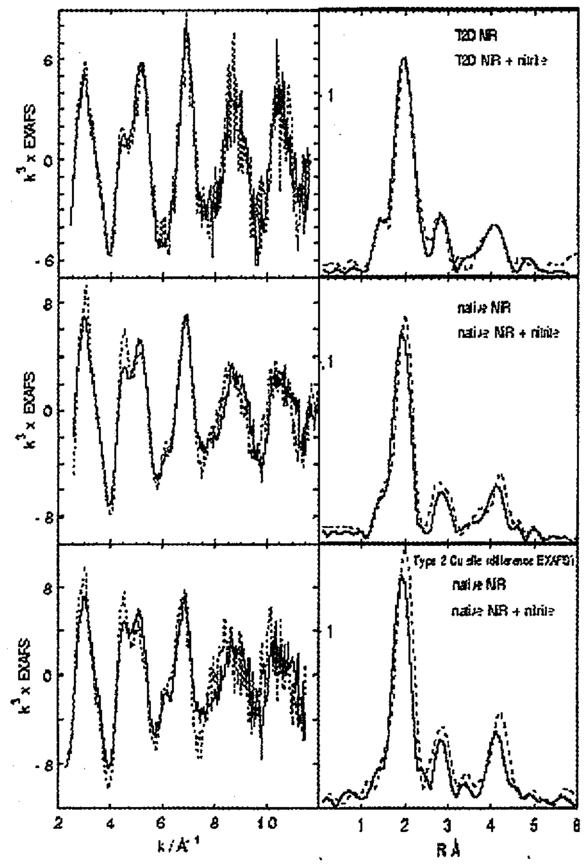

Figure 14: Refined EXAFS data for nitrite reductase.

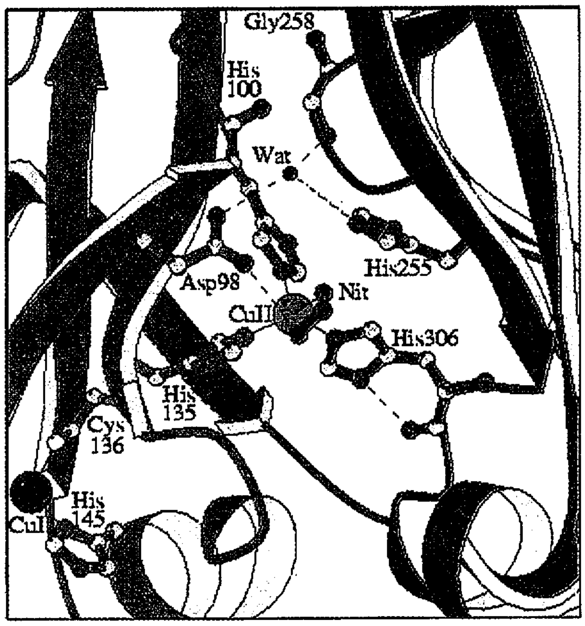

Figure 15: The catalytic copper active site in nitrite reductase, as revealed by a combination of EXAFS and protein crystallography.

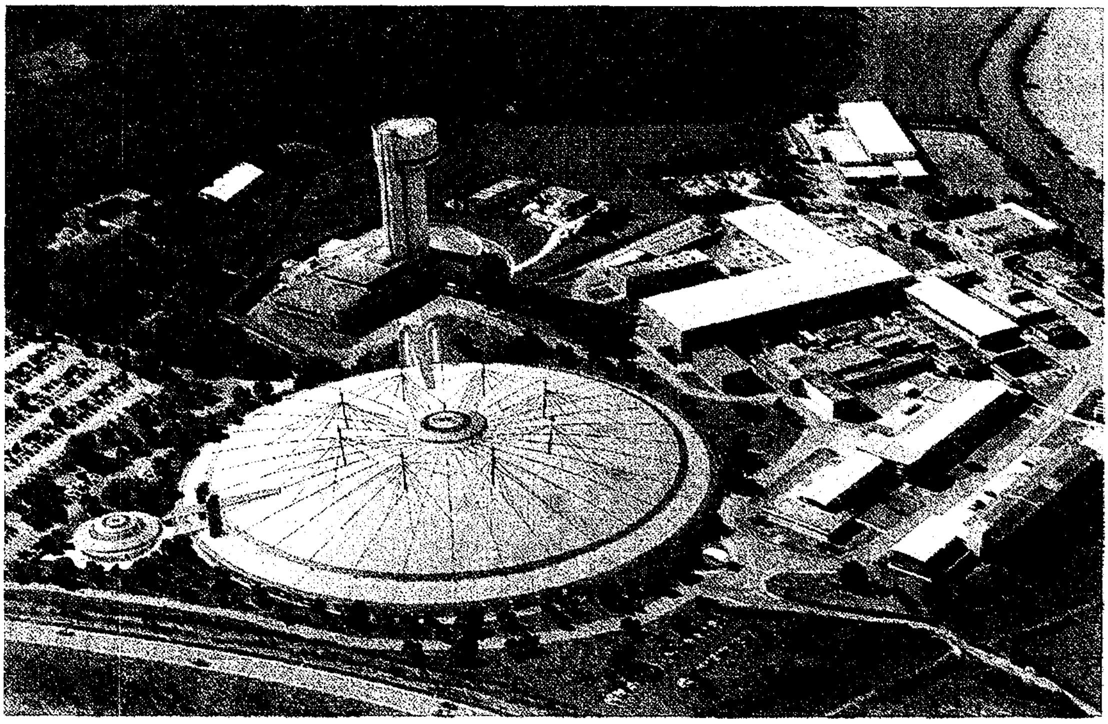

Figure 16: A computer-generated impression of how the projected DIAMOND facility will look at the Daresbury Laboratory, UK. Compare this with Figure 1.

widest context, as the service is flexible enough to be tailored to meet individual customer needs. Thus an experienced SR user can pay for raw data to be collected on a sample sent by post, or a new user can receive advice and consultation from the SR experts on experimental design, have an experiment carried out on their behalf, and have data processed and in some cases interpreted for them. This lowers the barrier to use of SR, by ensuring that a customer need not become an expert in the SR analytical techniques employed. Pre-allocation of time slots to industrial work ensures fast turnaround times, which are a requirement of the industrial working environment. The service is particularly aimed at companies who have never before considered SR use, perhaps through ignorance or inexperience, and so publicity and marketing has played a large part in establishing DARTS. Inquiries about the service have also been received from developing countries such as Nigeria, South Africa, Syria and Egypt, although as yet nobody in these or other developing countries have become customers.

This service-mode access to SR has also been extended to UK academia, through funding from the UK Engineering and Physical Sciences Research Council (EPSRC). Academics who are new to SR are being targeted, although applications are peer-reviewed. So, the word is being spread about SR.

Into the future ... and conclusions

As previously stated, many new SR sources are under construction or in advanced planning stages. SR sources are thought of in "generations", the latest machines being "third generation" sources, in that they provide radiation that has been "customized" by insertion devices such as the aforementioned wigglers and undulators. India, China, Taiwan, South Korea and Brazil now have SR facilities. Nearing completion is the Spring-8 facility in Japan, a Swiss Light Source has been approved for construction, and plans are well advanced for a new SR source in the UK. The new UK facility, DIAMOND (see figure 16), will be a replacement for the existing SRS, and complementary to the ESRF. This source and others will enable studies to be carried out on smaller samples, to higher degrees of accuracy and on shorter timescales than are possible with current facilities, and are bound to lead to "new science". Certainly, when SR was first used purposefully over thirty years ago, nobody could have dreamed of the possibilities that would open up and be realized in such a short span of time.

Patterns of SR use are also changing—extrapolating these changes into the future is possible up to a point. SR research was initially driven by physicists, looking into the most fundamental but perhaps least directly applied of problems. Over the years, materials scientists, chemists and ultimately biologists became involved, giving birth to interdisciplinary fields such as "molecular biophysics". One of SR's greatest strengths is its interdisciplinary nature, through which otherwise specialized scientists can learn from each other.

As larger sections of industry become more aware of SR and its applications, their use of SR and their priorities will become more influential. This is occurring already—the explosion in biological structure studies has piqued the interest of the relatively rich pharmaceutical industry. They have not been slow to recognize the potential value in such research, and their support for academia and, both directly and indirectly, SR work, is clearly shifting the centre of gravity of SR research towards the biological end of the spectrum. Applications in the more traditional materials area continue to grow strongly too—but growth is strongest in the life sciences.

So after over 30 years of staggering growth, SR techniques have come of age and are poised on the brink of new areas of applications, driven both by technological and commercial development. It is a most exciting field in which to be working, not merely from a scientific viewpoint but also from a commercial viewpoint. Just like synchrotron radiation itself, the future is very bright indeed.

Acknowledgments

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Business opportunities and alliances

Huff and Puff, but NIST makes sure the house stays up

As the Big Bad Wolf learned long ago, masonry buildings do not blow down. NIST's Building and Fire Research Laboratory wants to keep it that way. In the summer of 1997, the agency's Cement and Concrete Reference Laboratory (CCRL) launched three new programmes to help more than 150 building materials laboratories evaluate their quality testing methods for brick, masonry block and masonry mortar, respectively.

The CCRL, which is sponsored by the American Society for Testing and Materials, is sending both physical samples of the masonry material and testing instructions to the laboratories being evaluated. The laboratories perform their quality tests on the samples and return the results to NIST. The CCRL then checks the accuracy of the tests and ranks the results of all participating laboratories. Each laboratory receives a confidential statistical analysis showing how its performance fared in comparison to other participating, but unnamed, laboratories.

The CCRL devised the proficiency sample programs for masonry materials in consultation with the ASTM, the National Concrete Masonry Association and the Brick Institute of America. The new programs are in addition to existing CCRL proficiency sample programs for Portland cement, Portland cement concrete, blended cement, masonry cement and pozzolans.

For information on the CCRL and its proficiency sample programs, contact James H. Pipelert, A365 BuildingResearch Bldg., NIST, Gaithersburg, MD. (Source: *NIST Update*, 18 August 1997)

Proposals sought for precision measurement grants

NIST is seeking project proposals for two research grants for fiscal year 1999 in the field of precision measurement and fundamental constants. Each Precision Measurement Grant of $50,000 for one year may be renewed by NIST for up to two additional years for a total of $150,000. Prospective candidates must submit summaries of their proposed projects and biographical information to NIST by February 2, 1998.

NIST Precision Measurement Grants are awarded each year to faculty members of US universities or colleges for work in determining values for fundamental constants, investigating related physical phenomena or developing new, fundamental measurement methods.

Visit the Precision Measurement Grants World Wide Web page at http://physics.nist.gov/ResOpp/ grants/grants.html (Source: *NIST Update*, 18 August 1997)

D. PUBLICATIONS AND WEB SITES

Web Sites

Advanced Technology Corporation

Advanced Technology Corporation's (ATC) patented Stress-Strain Microprobe (SSM) system is recognized worldwide (1996 R&D 100 Award winner) as an innovative alternative to conventional, destructive methods of testing metallic samples.

For more information: Contact Fahmy Haggag, President, Advanced Technology Corp., 661 Emory Valley Road, Suite A, Oak Ridge, TN 37830-7751; Tel.: 423/483-5756; Fax: 423/481-3473; Web site: http://users.aol.com/haggagf/atc.htm. (Source: *Advanced Materials & Processes*, September 1997)

RJ Lee Instruments Ltd.

RJ Lee Instruments is "a different kind of SEM manufacturer". Providing sophisticated yet user-friendly analytical instruments to application-specific markets, its novel approach and ability to solve complex and varied materials analysis problems is the basis for its success.

Address: 515 Pleasant Valley Road, Trafford, PA 15085; Tel.: 412/744-0100; Fax: 412/744-0506. Web site: http://www.rjleeinst.com (Source: *Advanced Materials & Processes*, September 1997)

Web sightings

A description of World Wide Web sites that pertain to the NDT community. These listings make it easy to access the site. A brief description of the information to be found at the site is included.

MQS Inspection, Inc.

Stop and visit the MQS Web site for the latest on NDE and quality inspection services. Check out NDE training schedules.

MQS Inspection, Inc., 2301 Arthur Avenue, Elk Grove Village, IL 60007, Tel.: 1-800-638-5227, Fax: 847-981-9396, E-mail: mqs@execpc.com; Web site: http://www.mqsinspect.com

Panametrics

Visit our Web site. It is packed with features to help out with product selections, application information and theories. You will find abundant information on ultrasonic flaw detectors, thickness gauges, transducers, and scanning systems. An Application Section will be added to provide prospects with immediate feedback on cost-effective solutions. Web site: http://www.panametrics.com.

StressTel Corporation

StressTel Corporation offers a complete portable line of ultrasonic thickness gauges, corrosion gauges, precision gauges, and bolt stress measurement devices. For more specific detailed information and convenient catalogues on non-destructive ultrasonic testing equipment visit our Web site at http://www.stresstel.com

American Society for Nondestructive Testing (ASNT)

ASNT Web site is your Internet link to the NDT industry. E-mail the staff, catch up on Section activities, get the latest information on conferences and seminars. Plus a full-service bulletin board, exclusively for ASNT members. Preview important publications, locate Society officials, download the latest how-to articles. Web site: http://www.asnt.org (Source: *Materials Evaluation*, May 1997)

Publications

Nondestructive Testing Handbook

Nondestructive Testing Handbook, second edition; volume nine: Special nondestructive testing methods. Topics include: Acoustoultrasonics; Holography; Infrared thermography; Metal identification; Potential drop method; Tap testing; Vibration analysis; Metal identification; Magnetic resonance imaging.

To order, contact: American Society for Nondestructive Testing; Attn: Book Department, 1711 Arlingate Lane, P.O. Box 28518, Columbus, OH 43228-0518. Tel.: 800/222-2768 (USA only); or 614/274-6003. Fax: 614/274-6899. (Source: *Materials Evaluation*, January 1996)

Determination of Airborne Fibre Number Concentrations

Determination of Airborne Fibre Number Concentrations, by the World Health Organization Publications. Contents: Principle of the method; Scope of application; Specifications of parameters; Sampling; Evaluation; Counting and sizing fibres; Calculation of fibre concentration; Accuracy, precision and lower limit of measurement; Quality assurance; Static monitoring; Characterization of fibres, References.

Determination of Airborne Fibre Concentrations. A Recommended Method, by Phase Contrast Optical Microscopy (Membrane Filter Method). 1997, vii + 54 pages (available in English; French and Spanish in preparation). ISBN 92 4 154496 1. Sw.fr. 19.-/US$ 17.10. In developing countries: Sw. fr.13.30. Order number 1150446. Fax: (41 22) 791 48 57. E-mail: publications@who.ch. Order from the World Health Organization, Distribution and Sales, 1211 Geneva 27, Switzerland.

"Materials Selection & Design" Handbook provides common ground for designers, materials engineers

What every design engineer should know about materials selection (and what every materials engineer should know about design) can be found in the newest ASM Handbook, *Materials Selection and Design.*

Designers will benefit from a better understanding of the basic concepts, methodologies, and information sources available for dealing with materials selection and integrating it into the engineering design process.

Materials engineers will benefit by learning how to provide better input throughout the product design and development process, from initial concept through manufacturing. Detailed information about the various steps in the design process, including concept, configuration, and embodiment design are provided.

The Handbook's contents are divided into seven major subjects: The Design Process; Criteria and Concepts in Design; Design Tools; The Materials Selection Process; Effects of Composition, Processing and Structure on Materials Properties; Properties versus Performance of Materials; and Manufacturing Aspects of Design.

To order *Materials Selection and Design* (ISBN: 0-87170-386-6), contact the Member Services Center, ASM International, Materials Park, OH 44073-0002. Fax: 440/338-4634; E-mail: mem-serv@po.asm-intl.org. Price: $160 (ASM members: $128). For more information, contact the ASM Member Services Center or visit the ASM Website at http://www.asm-intl.org/. (Source: *Advanced Materials & Processes*, September 1997)

New Alloy Digest CD-ROM makes in-depth materials property data easier to find, use

For in-depth, proven data on nearly 4,000 worldwide metals and alloys—from chemical composition to detailed property and process information—design and materials engineers trust ASM's *Alloy Digest.*

Now, the new and improved *Alloy Digest CD-ROM, Version'96* makes more than 8,000 pages of data more accessible than ever.

Alloy Digest covers 36 materials groups, including aluminium, beryllium, carbon steel, cast iron, chromium, cobalt, copper, lead, magnesium, molybdenum, nickel, stainless steel, tantalum, titanium, tool steel, zinc and zirconium.

Another advantage of the CD-ROM versus the printed page is its ability to archive useful information.

For more information, contact the ASM Member Services Center, Materials Park, OH 44073-0002. Fax: 440/338-4634. E-mail: support@po.asm-intl.org. For more information, contact the ASM Member Services Center or visit the ASM Website at http://www.asm-intl.org/.

E. CALENDAR

Meetings

Sixth Annual Research Symposium on NDE Methods for Materials Characterization held from 17 to 21 March 1997, Houston, Texas

The Sixth Annual Research Symposium focused on methods for materials characterization with specific topics on:

- Ageing materials infrastructure
- Process control characterization
- Characterization of space materials
- Material properties characterization
- Industrial material characterization applications
- NDE of infrastructure.

For more information, contact: B. Boro Djordjevic, Center for NDE, 102 Maryland Hall, Johns Hopkins University, 3400 North Charles Street, Baltimore, MD 21218, Fax: 410/516-5293.

Conferences

1997

29 October-1 November
COMPOSITES '97. Joint conference of Composites Fabricators Association and Society for the Advancement of Material and Process Engineering, Orlando, FL. Contact: Charles Hamermesh, SAMPE Technical Director, P.O. Box 2459, Covina, CA 91722-8459; Tel.: (818) 331-0616, extension 602.

1998

22-23 January
International Symposium on Acoustic Emission: Standards and Technology Update, Ft. Lauderdale, FL. Contact: Sotirios J. Vahaviolos, Symposium Chairman, Physical Acoustics Corp., P.O. Box 3135, Princeton, NJ 08543; Tel.: (609) 844-3010; Fax: (609) 895-9726; E-mail: sotirios@pacndt.com

23-27 March
9th Asia-Pacific Conference on NDT, in conjunction with ASNT's Spring Conference and 7th Annual Research Symposium, Anaheim, CA. Sponsored by ASNT. Contact: ASNT.

30 March-2 April
Structural Materials Technology: An NDT Conference, San Antonio, TX. Co-sponsored by FHWA and TXDOT. Contact: Ronald Medlock, Materials and Tests Div., Texas Dept. of Transportation, 125 E. 11th St., Austin, TX 78701-2489; Fax: (512) 465-7616; E-mail: rmedloc@mail.gov.dot.state. tx.us

15-17 April
INALCO '98, 7th International Conference on Joints in Aluminum, Cambridge, UK. Contact: Rosemary Cook, Conference Organizer, TWI, Abington Hall, Abington, Cambridge CB1 6AL, UK; Fax: +44 (0) 1223-894363; E-mail: meetings@twi.co.uk

3-6 June
8th European Conference on Composite Materials (ECCM-8), Naples, Italy. Contact: Crivelli Visconti, Chairman, ECCM-8, DIMP—University of Naples, Piazzale V. Tecchio, 80125 Naples, Italy. Fax: +39 81 7614212; E-mail: crvisco@unina.it; Web site: http://www.eccm98.etruria.net

15-19 June
6th International Conference on Welding and Melting by Electron and Laser Beams (CISFFEL 6), Toulon, France. Contact: Institut de Soudure, ZI Paris Nord II, 90 Rue des Vanesses, 93420 Villepinte, France. Tel.: +33 1 49 90 36 00; Fax: 33 1 49 90 36 50.

1999

4-13 August 1999
XVIII International Union of Crystallography Congress, Glasgow, Scotland, UK.
Contact Gill Houston, Northern Networking Ltd., Congress Central Office, Bellway House, 813 South Street, Glasgow G14 0BX, Scotland, UK. Tel.: +44 (0) 141 954 4441; Fax: +44 (0) 141 954 2656; E-mail: gill@glasconf.demon.co.uk; Web site: http://www.chem.gla.ac.uk/iucr99

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