NPL REPORT
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EXECUTIVE SUMMARY

This report describes the work completed under NPL’s focussed programme of work under ‘Representation of UK Industry on International Materials Standards MII 2.3’. The work described in this report was conducted over the period April 2002 to March 2005. As the development of standards from new work item to publication can take years, there is no simple chronological break in the work; this report covers the full duration of the work programme.
National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

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Approved on behalf of the Managing Director, NPL,
by Dr M G Cain, Knowledge Leader, Materials Processing Team
authorised by Director, Engineering and Process Control Division
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1 INTRODUCTION

UK industry looks to NPL to provide informed and independent expertise on International and National Standards bodies and other related industrial committees. This type of activity has been recognised as an important dissemination route for work areas funded by DTI Programmes. The work described under ‘Representation of UK Industry on International Materials Standards MII 2.3’ is a logical progression of the work in previous DTI Programmes that is not currently funded; it exploits the internationally renowned expertise of NPL, which has accumulated over the years. It ensures that this expertise is fully utilised in providing a clear dissemination route for this knowledge into International Standards that are for the benefit of UK industry competitiveness, quality and environmental impact.

The drafting of these standards is acknowledged to be a protracted exercise, which normally extends years beyond the lifetime of the DTI NMS Programme that supported the original underpinning knowledge or permitted the development of new test methods.

The standard areas covered by MII2.3 are a reflection of the fields of internationally renowned expertise present at NPL. The activities covered under MII2.3 cover a wide range of areas including:

- General engineering of metals for automotive, power generation, petrochemical storage and transport, and construction
- Healthcare products for an ageing society
- Metal product manufacture
- Machinery
- Aerospace
- Chemical products

Many of the standards developed are generic methods for characterising a broad spectrum of materials to generate data for component design, or for manufacturing quality control issues.

This project funded senior staff to attend meetings convened by the International Standards bodies such as CEN, ISO and ASTM, and those covered by BSI. Staff attended ISO and CEN meetings in the capacity of UK delegates, reflecting their UK industrial sectors’ positions on these standards. This meant that UK industrial interests were represented in European and global contexts, so that the standards developed were not detrimental to UK industrial sector outlooks by limiting their competitiveness or by forcing them to rely on difficult to obtain (on a national level) reference materials or devices. The Standards organisations rely on their membership at the committee level to propose direction and scope of standards in their fields of expertise.

2 SCOPE OF IMPACT REPORT

This report is structured in two sections:
- Part One – Case Studies
- Part Two – Summary by Subject Areas
The objectives of MII2.3 were:

- To support UK industry through the development of appropriate National, European and International Standards by providing independent representation on Standards Committees and providing assistance in drafting of standards.
- To ensure that UK industry is informed of current developments in National and International Standards in the Materials sector, which are likely to affect industrial competitiveness.
- To defend the UK position on standards that do not accord with practices followed by UK industry.
- To formulate and implement key pathways for the dissemination of relevant National and International standards activities to UK-based stakeholders.
- To maintain and ensure DEPC’s (incorporating MATC) visibility as a key player in International materials metrology.
- To develop nanoscale standardising opportunities.
- To promote and support discussion of nanoscale-related metrology issues through an International Workshop.

The project resulted in the involvement of NPL in over 30 Standards and associated industrial committees and technical input into 70 documentary Standards. NB. The term ‘standards committee’ is a generic one; it also applies to sub-committees and working groups.

This report is a dissemination tool to promote the impact that these standards provide on industrial competitiveness, quality of life and the environment.

2.1 CASE STUDY 1- BIOMATERIALS STANDARDS

F2312-04: Standard Terminology Relating to Tissue Engineered Medical Products
F2211-04: Standard Specification for General Classification for Tissue Engineered Medical Products

The cost of maintaining the increasingly aging population of Western societies, where individuals are living for longer periods of time and expect a high quality of life, is considerable to health service providers.

These factors, coupled with a lack of compatible tissue and organ donors has led to the development of Tissue Engineered Medical Products (TEMs), as potential replacements to ‘worn-out’ organs avoiding the long-term immuno-suppressant drug regimes as TEMs consist of the patient’s own cells grown on a support matrix.
Figure 1: A polycaprolactone tissue scaffold. Tissue scaffolds are typically complex porous materials consisting of a network of interconnected pores.

As TEMPs are regarded as medical devices the FDA requires standards for TEMPs for regulatory purposes. These standards are necessary to define the nature of materials used for scaffolds and to provide methods for the measurement of their efficacy.

The development of new TEMPs along with further application of existing materials requires accurate measurement of cell adhesion, growth and specialisation within biomaterial scaffolds. These biomaterial scaffolds may be constructed of a range of different materials such as metal, ceramic or polymer. Cells used to seed these scaffolds have very specific morphology and environmental requirements. Therefore, the first standards addressing these regulatory needs under this project have been those concerned with accurate characterisation methodologies for these scaffolds.

2.2 CASE STUDY 2 - PLASTICS STANDARDS

ISO 6721-11: Determination of physical dynamic properties

One of the key measurements to determine the suitability of a plastic/polymer for an application is its Glass Transition Temperature. This is the temperature at which a polymer reversibly changes from a rubbery or viscous texture to a hard and comparatively brittle condition. Plastic components are present in almost every modern device from power cables and aeroplanes to PC disks and data storage devices. Plastic components in close proximity to heating or cooling elements need to withstand changes in temperature to remain ‘plastic’ under all conditions of usage. For example,
the plastic sheath around conductive wires needs to remain rubber-like to ensure safe operation, even though the current passing through the wires causes localised heating. Due to the widespread use of many plastics as insulating material for power cables, there is a need to accurately measure the temperature at which their visco-elastic properties are lost because they undergo a phase transition to a more brittle form. When this transition occurs it is much more likely that the plastic may become damaged and cease to function with a significantly increased risk to users of devices with power cables.

The measurement of the Glass Transition Temperature (Tg) of a plastic is not straightforward. It can change remarkably due to the test conditions such as the rate at which it is heated and the observation time. Over the years, a number of measurement standards have been adopted to accurately measure the Tg of polymers (including composites). These include standard test methods for Thermal Analysis, Thermomechanical Analysis, Dynamic Mechanical Analysis (DMA) and Differential Scanning Calorimetry (DSC).

Figure 2. Dynamic Mechanical Analysis of plastic composites is vital to many leading UK manufacturers and users of polymeric materials.

The most widespread methods used are DSC and DMA. These techniques are increasingly used for material qualification because of the ease of sample preparation and speed of data generation. However, the data generated can vary depending on the technique and measurement methods used. The Tg is often difficult to observe in DSC, whilst in DMA it is easier to observe.

A prototype temperature reference specimen has been produced, consisting of indium bars embedded in a carbon fibre laminate for use in DMA (Figure 3). This prototype is part of the calibration procedure within the new ISO standard for Tg by DMA, ISO6721-11.
Currently, it appears that most DMA results are incorrect due to temperature lag effects, which causes the measured data to vary with heating rate. This reference spectrum can be used as part of an instrument calibration procedure to identify temperature lag effects and which heating rates should be used to establish the true Tg.

This standard will help in the development of controlled cured polymers at the manufacturing and quality control process stage.

Dissemination of this work has been through its inclusion as a technical annex in ISO 6721-11 and through NPL measurement note DEPC(MN)17, which are widely distributed to industrial end-users, e.g., through the Industrial Advisory Group.
3. IMPACT OF NPL STAFF ON THE STANDARDS MAKING AND REVISION PROCESSES, BY SUBJECT AREA

The contribution (i.e. impact) that NPL staff have made to the standards making and revision processes, as funded by the MII2.3 project are dealt with, by subject area, below.

3.1 PLASTIC STANDARDS

(Dr. G. Dean)

Dr. Dean has provided technical input into the standards presented to the following working groups of the ISO TC61 Committee:

SC2/WG1 Static Properties
SC2/WG4 Dynamic Properties
SC2/WG7 Fracture and Fatigue Properties
SC2/WG8 Forms of Data Presentation
SC11/WG5 Polymeric adhesives

**SC2/WG1: Static Properties**

Technical input was provided by Dr Dean into the following standards:

ISO/CD 18872: Determination of tensile properties.
ISO 527-1 and 527-2: Determination of tensile properties.
NWIP: Dynamic tensile characterisation of plastics.

**SC2/WG4 Dynamic Properties**

ISO 6721-11: Determination of glass transition temperature.

This is discussed in more detail in Case Study 2.

ISO 75: deflection temperature under load.
NWIP: Determination of dynamic properties: Compressive vibration method.

**SC2/WG7 Fracture and Fatigue Properties**

NWIP: Determination of fracture toughness of ductile polymers using a J-integral method.

**SC2/WG8 Forms of Data Presentation**

Dr Dean provided technical input into the following standards under MII2.3:

NW1 10350-3: Presentation of comparable single data points: Part 3 Thermoplastic elastomers.
ISO 11403-3: Presentation of comparable multipoint data: Part 3 Environmental influences on properties.

**SC11/WG5: Polymeric Adhesives**

Under MII2.3 Dr Dean has represented the UK industrial interests in the development of the following standards and work items:

ISO/CD 17194 Adhesives: a standard database  
ISO/CD 19212 Determination of the temperature dependence of adhesives.  
ISO/DIS 21368: Guidelines for the fabrication of adhesively bonded structures.  
ISO/FDIS 14678: Determination of resistance to flow.  
NWIP: determination of the mode 1 adhesive fracture energy of structural adhesives using double cantilever beam and tapered double cantilevered beam species.

3.2 TECHNICAL CERAMICS/ORTHOPAEDIC IMPLANTS  
(De R Morrell)

The overall driver for the ceramics manufacturer is to be able to provide traceable data on products by recognised test methods appropriate to the product. The overall driver for the user is to have confidence that the methods are not producing unambiguous or irrelevant results when applied to a component or device, as opposed to test-piece data.

**ISO TC206: Fine Ceramics**

Dr Morrell has contributed to 20 standards on fine ceramics in the period covered by MII2.3, and has been the UK delegate on the ISO TC206 panel. In support of the UK delegation to ISO TC206, Dr. Morrell has made a full contribution to deciding the UK’s policy through attendance at BSI Committee RPI/13 meetings.

Four of these standards have been published within the lifetime of MII2.3:

ISO 20808: 2004: Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of friction and wear characteristics of monolithic ceramics by ball-on-disk method.

A further 16 standards or work items are subject to approval:

ISO NP 34: Fine ceramics (advanced ceramics, advanced technical ceramics)- Test methods for determination of fracture toughness of monolithic ceramics - single edge vee-notch beam (SEVNB) method.

Dr. Morrell is the convenor of the above work item.
ISO/DIS 18755: Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of thermal diffusivity of monolithic ceramics by laser flash method.
ISO/DIS 20502: Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of adhesion of ceramic coatings by scratch testing.
ISO/DIS 20505: Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for interlaminar shear strength of continuous fibre-reinforced composites at room temperature by the double-notched test pieces and Iosipescu test.
ISO/DIS 20506: Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for in-plane shear strength of continuous fibre-reinforced composites at room temperature by the Iosipescu test.
ISO/DIS 24369: Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of content of coarse particles in ceramic powders by wet sieving method.
ISO/DIS 24370: Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for fracture toughness of monolithic ceramics at room temperature by chevron notched beam (CNB) method.
ISO/CD 17092: Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of corrosion resistance of monolithic ceramics in acid and alkaline solutions.
ISO/CD 18452: Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of thickness of ceramic films by contact probe profilometer.
ISO/CD 22214: Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for cyclic bending fatigue of monolithic ceramics at room temperature.
ISO/CD 22215: Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for tensile creep of monolithic ceramics.
ISO NP 35: Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of tap density of ceramic powders.
NWIP 22197-2: Photocatalytic materials.

3.3 FATIGUE TESTING
(Dr F Kandil)

Summary

Representation on these committees ensures that NPL’s expertise continues to be disseminated directly to the relevant focus groups at the same time as maintaining UK profile. Feedback is provided directly from industry concerning their immediate measurement problems and advice is frequently proffered concerning solutions.

NPL continues to be very active in the standards activities in the fatigue testing area. Fatigue testing is performed by so many companies, large and small, in a wide spectrum
of industries ranging from small test houses to large power or aerospace corporations. These look to NPL’s experience to represent their interests in the formulation of new and improved standards.

Activities undertaken by Dr Kandil during the period of this report were to represent UK industrial interests in the development of new standards in the areas of fatigue testing and measurement uncertainty. Representation within BSI, CEN, ISO and ASTM are described.

**ISO/TC164/SC5: Fatigue testing**

Dr Kandil is a long-standing UK representative on this committee and has provided representation at each annual meeting, including a number of presentations. Detailed contribution was provided to a number of standards including:

ISO 12106:2003 ‘Metallic materials – Fatigue testing - Axial strain controlled method’
BS ISO 12107:2003 ‘Metallic materials – Fatigue testing – Statistical planning and analysis of data’

**ISO/TC164/SC5/WG1: General Principles - Fatigue**

Dr Kandil was the convenor of this project, producing a new draft standard. It was agreed by the committee that this standard should include as far as possible, aspects from all standards developed by this committee.


The above project was also initiated by Dr. Kandil. A new ISO standard has been drafted, ‘Fatigue materials- Fatigue testing- Alignment of fatigue testing machines’. This standard is based on work carried out at NPL in collaboration with EU and VAMAS groups.

**ISO/TC164 AD HOC Group on Terminology**

Reviews of the latest standards documents were made by this group, which included Dr. Kandil as the ISO/TC164/SC5 representative.

**ASTM**

Dr Kandil is a member of an international forum that is currently revising ASTM standards on ‘Fatigue and Fracture’ and ‘Mechanical Testing’ (dealt with by Committees ASTM E08 and E28). These standards are widely used by industry in the UK in several types of mechanical tests and NPL’s involvement ensures that UK industrial interests are not unfairly disadvantaged in winning international business, particularly from USA aerospace and power generation industries. Dr Kandil (as chairman of both the ISO/TC164/SC5/WG11 and VAMAS/TWA13) is also keen that there will be no conflict between the ISO/ASTM/VAMAS Documents.
3.4 MICROPROBE ANALYSIS  
(Dr S Saunders)

Many materials have complex microstructures, the chemical and structural aspects of which crucially determine performance. Thus measurement methods are required to accurately describe these micro features, and electron-probe microanalysis (EPMA) is widely used in industry, test houses and research organizations to determine the chemical composition of areas only a few microns in diameter. The driver for working on the ISO standards is to ensure that they reflect more closely actual practice in this area; Chinese and Japanese output in this area is considered to be too prescriptive in terms of machines and reference materials and the standard of English in the relevant documents is poor. The NPL input through the BS CII/9 committee is for example ensuring that UK-produced reference materials are acceptable in a global market and that the practices for determining magnification in the SEM are not reliant on commercial objectives of the USA.


The UK delegation has driven the microbeam standards, which is seen as a high priority in microprobe analysis. The lack of a terminology standard has slowed progress in this area, though this has been addressed by Dr Saunders in his work under MII2.3. During the period covered by MII2.3 six microbeam standards have been published with a further 5 standards subject to panel approval.

These standards are used by UKAS assessors when auditing accredited analysis laboratories. This keeps quality at a high level and hence their customers can be assured of the correctness of results. When drafting these standards care was taken that the UK laboratories would be able to implement the standards and that they would not be disadvantaged by having to use US or Chinese reference materials, for example.

The following standards have been published:

BS ISO 17470: Electron Probe Microanalysis-Guide To Qualitative Point Analysis By Wavelength Dispersive X-Ray Spectrometry.  
BS ISO 22029: EMSA/MAS Standard file format for Spectral Data Exchange.

UK input into a number of other projects was provided by Dr Saunders under MII2.3. The following projects have resulted in draft standards that are subject to approval:

ISO DIS 23833: Vocabulary
ISO/WD 17270: AEM – Experimental Parameters for EELS.

3.5 GAS CYLINDERS /UNIAXIAL TESTING
(Mr M S Loveday/ Mr. G. Sims)

Approval of new types of Gas Cylinder needs to be in accordance with the EC 1984 Directive, under contract from the Heath and Safety Executive; as such NPL has statutory obligations in this field. NPL remained a ‘paper’ member of the appropriate BSI committees in order to maintain a watching brief on Standards development.

Uniaxial testing of metallic materials provides the fundamental mainstay of material property data that pervades throughout all sections of society where materials are used. Tensile test data, together with Hardness and Charpy Impact are used for product release certification for virtually all metallic materials, which are subsequently used in all industrial sectors. The data are also used for design of components and the subsequent life monitoring of plant in safety critical applications, particularly in the aerospace and power generation industries.

During MI12.3 a total of fourteen standards on gas cylinders had technical input provided by Mr Loveday and Mr Sims.

EN 849: Transportable gas cylinders - Cylinder valves :Specification & type testing.
EN 962: Transportable gas cylinders - Valve caps design, construction and tests.
EN ISO 11114: Transportable gas cylinders - Compatibility of cylinder and valve materials with gas contents.
EN ISO 13341: Transportable gas cylinders- Fitting of valves to gas cylinders.
PrEN 12205: Transportable gas cylinders- Non Refillable metallic gas cylinders.
PrEN ISO 11439: High pressure cylinders for the on-board storage of natural gas as a fuel for automotive vehicles.
PrEN 1975: Transportable gas cylinders- Specification for the design and construction of refillable transportable seamless aluminium alloy gas cylinders of capacity from 0.5 l up to 150 l.
PrEN 12862: Transportable gas cylinders- Refillable welded aluminium alloy gas cylinders.
PrEN 12245: Transportable gas cylinders- fully wrapped composite cylinders.
PrEN 12257: Transportable gas cylinders - Seamless, hoop wrapped composite cylinders.
ISO 7866: Gas cylinders- Refillable seamless aluminium alloy cylinders-Design, construction and testing.
BS EN: Non-refillable metallic cartridges for liquefied petroleum gases, with or without valve, for use with portable appliances Construction, testing and marking.
**ISO TC164 SC1 WG4 Tensile Testing**

Mr Loveday has made technical input into the following standards during MII2.3:

ISO 6892: Tensile testing standard
BS 1610 Pt1: Force Verification of Testing Machines : Compression
BS1610 Pt 3: Load Calibration of Lever Creep Machines
BS EN 10002 Pt1: Tensile Testing of Metallic Materials
BS EN 10002 Pt2: Load Calibration of Tensile Testing Machines
BS EN 10002 Pt3: Verification of Force Proving Devices
BS EN 10002 Pt4 : Calibration of Extensometers
BS EN 10002 Pt5: Tensile Testing of Metallic Materials at Elevated Temperatures
Pr EN 10291: Uniaxial Creep and Rupture Testing at Elevated Temperatures
ECO 001046 : Stress Relaxation Testing
ECO 001040: Conversion values for steel hardness
ECO 001041: Hardness-Tensile strength conversion values for steels
ISO 204: Tensile Creep Testing
ISO 376: Verification of force proving devices
ISO 783: High Temperature Tensile Testing
ISO 6892: Tensile Testing of Metallic Materials
BS EN 10045 -1:1990: Charpy Impact test  on metallic materials
BS EN 10045 -2: 1993: Method for verification of impact testing machines
ISO/DIS 14556: Steel - Charpy V-notch impact test - Instrumented test method

3.6 IMPACT TESTING

(Mr M S Loveday)

After the hardness test, Charpy impact testing is the most popular test in industry. According to the latest data about 1.2 MECU is spent on the impact testing of metallic materials in the Europe for product release certification, covering a wide range of materials which are used in the manufacturing, automotive, power generation, nuclear and biomedical industries. The use of this test technique is continually evolving: instrumented impact testing of metallic materials has recently been automated in several steel plants in Japan, Sweden and the US and significant effort has been devoted in Japan to the development of fracture parameters from Charpy data. This has the potential to replace expensive and time-consuming fracture toughness tests.

Mr. Loveday has represented the UK in the development of ISO standards for Charpy impact testing. These include the standards listed below:

BS 131 Pt 6 :1998: Notched bar tests : method for precision Charpy testing
BS 131 Pt 7 :1998: Notched bar tests Verification of the test machine
BS EN 10045 -1:1990: Charpy Impact test on metallic materials
BS EN 10045 -2: 1993: Method for verification of impact testing machines
ISO/DIS 14556: Steel - Charpy V-notch impact test - Instrumented test method
3.7 POLYMER DEGRADATION

(Dr A S Maxwell)

Environmental degradation accounts for the majority of in-service failures of plastic components. In many cases, these failures are in comparatively low cost products in non-demanding applications. However, plastics are increasingly being used in far more exacting applications. In the oil and gas industry, nylon is now being used in flexible multi-layer pipelines to carry crude from the seabed. In the chemical and water industries plastic liners are being extensively used for pipelines and vessels and in the aerospace industry environmental degradation necessitates the early replacement of thousands of polycarbonate aircraft windows every year.

Dr. Maxwell is a long-standing UK representative on ISO/TC61 committee on the ageing and degradation of polymers. The committee is to develop standards for the long-term performance of plastics in the environment. The sub-committee is divided into three main working groups:

- WG2 covering ultra violet light exposure
- WG3 covering chemical exposure
- WG7 covering basic test conditions and absorbance/loss of liquids

**ISO/TC 61/SC6/WG2 Polymer degradation – Light Exposure**

Standards proposed, reviewed and revised by this committee included:

- ISO 877: Plastics – Methods of exposure to direct weathering, indirect weathering using glass-filtered daylight and indirect weathering by daylight using Fresnal mirrors.

Detailed input reflecting the UK industrial position was provided by Dr. Maxwell.

**ISO/TC 61/SC6/WG3 Polymer degradation – Chemical Exposure**

Standards proposed, reviewed and revised by this committee included:


ISO DIS 22088 – Part6: Slow strain rate method is a new standard led by Tony Maxwell.

**ISO/TC 61/SC6/WG7 Polymer degradation – Basic Standards**

The following standards and new work items were reviewed by the above WG, with UK representation from Dr. Maxwell.

ISO 291: Plastics – Standard atmospheres for conditioning and testing.

ISO/TC61/SC2/WG2 Hardness and Surface Properties

Input was provided to the following standards by Dr. Maxwell:

ISO DIS 21509: Plastics and Ebonite – Verification of Shore Hardness Durometers.

4 DISSEMINATION ACTIVITIES

The MII2.3 project proposal outlines certain dissemination deliverables. These have been complied with as follows:

4.1 MII2 D3

A poster presentation was given at the EU metrology event, Warsaw Nanotechnology Workshop, with a summary report on the outcome.

4.2 MII2 D1

A presentation was made at the mid-term review of MII2.3 by Mr. John Sillwood, Project Leader.

4.3 OTHER DISSEMINATION ACTIVITIES

Dr. Kandil prepared and delivered a presentation titled "VAMAS inter-laboratory studies in LCF at elevated temperatures: an overview", at the ASTM meeting in Tampa, Florida 17-20 Nov 2003.

Dr. Kandil also responded to over 70 technical enquiries concerning fatigue standards, fatigue testing, and measurement uncertainty.

Measurement notes promoting the new standards published as are result of MII2.3 have been published and distributed to the relevant Industrial Advisory Groups.