Comité international des poids et mesures

Evolving Need for Metrology in Material Property Measurements

Report of the CIPM *ad hoc* Working Group on Materials Metrology (WGMM)

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Bureau international
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Organisation intergouvernementale
de la Convention du Mètre

AD-HOC WORKING GROUP ON MATERIALS METROLOGY (WGMM)

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SUMMARY

Over the last few years, the need for wider international collaboration in the measurement of materials properties has been under discussion at meetings of the Versailles Project on Advanced Materials and Standards (VAMAS). VAMAS approached BIPM (Bureau International des Poids et Mesures) with a view to including materials metrology in the formal international measurement structure established under the Metre Convention. Following a workshop at the BIPM in February 2005 the CIPM established an *ad hoc* Working Group on the metrology applicable to the measurement of material properties. This report details the work, findings and recommendations of the Working Group.

The working group has assessed a wide range of materials properties, studying in particular the need for improved traceability, better comparability of data and the availability of appropriate reference materials, all of which are important for regulators, manufacturers, standards bodies and accreditation bodies. Reference materials, in particular, play an important role in enabling traceability, in allowing calibration of test equipment and in ensuring confidence in data. The working group identified a number of different classes of materials that would merit consideration and a wide range of properties that are routinely measured. Some of these are already the subject of study by the CIPM's Consultative Committees and their Working Groups, notably hardness (in a working group of the CCM) and a range of thermophysical properties in a working group of the CCT (i.e. WG9).

The mutual recognition arrangement (MRA) drawn up in 1999 by the CIPM seeks to provide "a secure technical foundation for wider agreement for measurements related to international trade, commerce and regulatory affairs". This is normally achieved through international comparisons of measurements performed at NMIs and Designated Institutes to provide an independent assessment of performance. A small number of these comparisons have involved measurements of materials properties, including hardness, thermal properties and electrical conductivity, and the Working Group considered that these should be extended to include a wider range of properties.

At the first meeting of the Working Group, separate task groups were established to investigate materials properties measurement in five defined areas: mechanical, thermophysical, microstructural, functional and electrochemical. Each task group prepared an inventory of materials properties and these were reviewed to identify specific needs for better comparability and improved traceability. The definition of traceability also raises a number of issues when applied to the measurement of materials properties and these are discussed in this report.

The Working Group has made a number of recommendations aimed at ensuring an ongoing dialogue between VAMAS and the CIPM. As well as proposing joint activities, underpinned by a Cooperation Agreement, which would require VAMAS to report annually to the CIPM on needs for improved materials metrology, there are recommendations which will extend the work of the CIPM's Consultative Committees to ensure that CMCs for a number of important materials properties are included in the Key Comparison Database in due course.

These recommendations will, if implemented, address the issues, which limit the accuracy and repeatability of materials properties measurement and establish traceability in this vitally important field.

The recommendations are also given below.

RECOMMENDATIONS

TO CIPM

1 The Working Group recommends that the CIPM should sign a Cooperation Agreement with VAMAS in order to ensure an ongoing dialogue and actions with a view to identifying key traceability issues affecting the accuracy and repeatability of the measurement of materials properties.

2 The Working Group recommends that the CIPM should instigate a further review in 3 or 4 years time to evaluate the progress made and determine what further action, if any, is required.

TO CONSULTATIVE COMMITTEES

The Working Group recommends that CC working groups should be established to stimulate comparisons, establish measurement capabilities in NMIs and identify suitable certified reference materials with known uncertainties.

3 The Working Group recommends that the CCEM should establish a working group on electromagnetic properties of materials.

4 The Working Group recommends that the CCAUV should establish a working group on acoustic properties.

5 The Working Group recommends that the CCM and CCL should consider the case for a joint working group on mechanical properties of materials, with VAMAS representation.

6 The Working Group recommends that materials WGs established by CCs should encourage participation of all important stakeholders, including ISO/IEC, ILAC and VAMAS.

TO NATIONAL METROLOGY INSTITUTES

7 The Working Group recommends that NMIs should support materials metrology in their work programmes in order to implement and disseminate best practice in the measurement of materials properties.

8 The Working Group recommends that NMIs should encourage their staff to participate actively in the work of joint BIPM/VAMAS materials working groups.

To VAMAS

9 The Working Group recommends that VAMAS Steering Committee should initiate a top-down review, with other stakeholders, to identify priority actions in selected areas and draw these to the attention of CIPM, ILAC and NMIs.

10 The Working Group recommends that VAMAS develop with CIPM appropriate pilot studies (see Annex A for examples).

CONTENTS

ABBREVIATIONS

ANMET - APEC Network for Materials Evaluation Technology
APEC - Asia-Pacific Economic Community
BAM - Bundesanstalt fur Materialforschung und Prufung, Germany
BEV - Bundesamt für Eich- und Vermessungswesen, Austria
BIPM - Bureau International des Poids et Mesures
CENAM - Centro Nacional de Metrologia, Mexico
CIPM - Comité International des Poids et Mesures
CRM – Certified Reference Material
EIMRA - European Industrial Research and Management Association
ILAC - International Laboratory Accreditation Cooperation
IRMM - Institute for Reference Materials and Measurements, Joint Research Centre, EC
KRISS - Korea Research Institute for Standards and Science, Korea
LNE – Laboratoire National de Métrologie et d'Essais, France
NIST - National Institute for Standards and Technology, USA
NPL - National Physical Laboratory, UK
NMSD - National Measurement System Directorate, UK
OIML - International Organisation of Legal Metrology
PTB - Physikalisch-Technische Bundesanstalt, Germany
RM – Reference Material
SMU - Slovak Metrology Institute, Slovakia
VAMAS - Versailles Project on Advanced Materials and Standards
VNIIM - D. I. Mendeleyev Institute for Metrology

1 INTRODUCTION

This report details the work, findings and recommendations of an *ad hoc* Working Group on the metrology applicable to the measurement of material properties, or more succinctly "materials metrology" (WGMM). The WG was established by the CIPM (Comité Consultatif des Poids et Mesures) following international discussion of the metrology issues in this field raised by the VAMAS (Versailles Project on Advanced Materials and Standards) G7 pre-normalisation initiative in October 2005. The Working Group has assessed a wide range of materials properties; studying particularly the need for improved traceability, better comparability of data and the availability of appropriate reference materials. This report was tabled at the 2007 CIPM annual meeting in line with the Working Group's terms of reference. The work has already raised the profile of materials metrology internationally and engaged the leading NMIs (National Measurement Institutes) in recognising and addressing known difficulties in demonstrating traceability of many material properties to the SI.

The background to this study is given in Section 2, followed in Section 3 by the Terms of Reference; the material scope of the study; a discussion of the nature of intrinsic and procedural properties; and the existing materials work in Consultative Committees.

In Section 4 trends in metrology in BIPM/CIPM and EURAMET are described, together with the relevance of the Mutual Recognition Arrangement. Definitions in the VIM and for materials metrology are discussed in Section 5. Section 6 looks at the importance of materials measurements for innovation, reliability and safety, and reviews future needs identified in a UK study. The role and availability of reference materials is reviewed in Section 7.

The detailed work undertaken by the Task Groups is described in Section 8, with summaries of the findings in Sections 9-13. Finally, Section 14 addresses the issues identified regarding measurements, traceability and standards. Following a discussion of the issues in Section 15, conclusions and recommendations are presented in Section 16.

Suggestions for potential pilot studies are given in Annex A.

Meetings of the working group were held at:-

- NPL – May 2006
- NIST – December 2007
- LNE – May 2007 (immediately prior to VAMAS 2007 SC Annual Meeting).

Membership of the Working Group was widespread and included representatives from,

BAM	CENAM	INMETRO	NMIJ	VNIIM
BIPM	NMISA	IRMM	LNE	PTB
CCQM	ILAC	KRISS	NIST	
CCM	IMGC	NIMS	NPL	

There were multiple attendances from some organisations and some members also represented VAMAS.

2 BACKGROUND

The topic of "materials metrology" or "metrology applied to materials property measurement" has been discussed several times over the last decade in, for example, the collected papers edited by NPL in 1995 [1] and the NIST handbook published in 2003 [2]. It concerns the application of measurement science to the accurate and fit-for-purpose determination of material properties throughout the material's life-cycle. It includes the provision of reference materials where they perform an essential role for traceability and calibration aspects. The extra dimension compared to SI related measurements is that while many properties are "inherent" or "intrinsic" (e.g. density, modulus), in other cases the property measured is dependent on the test procedure. Hardness is one of the best examples of a "procedural" property. Hardness is a measure of the material's resistance to plastic deformation by indentation. It's value is largely dependent on how the indentation is imposed on the material.

Over the last few years, the need for wider international collaboration in the measurement of materials properties, and in particular the issues of standards and traceability have been under discussion at meetings of the Versailles Project on Advanced Materials and Standards. VAMAS operates under a Memorandum of Understanding signed by senior representatives of government in countries of the Economic Summit (G7) and of the European Commission. It supports international trade through projects aimed at providing the technical basis for drafting codes of practice and specifications for advanced materials. The scope of this international collaboration embraces all aspects of enabling science and technology required as a precursor to the drafting of standards. Through its activity, VAMAS fosters the development and harmonisation of international standards for advanced materials by the various existing standards agencies. Since its inauguration in 1982, VAMAS has had a considerable impact on the development of internationally accepted standards for engineering materials. The specification of materials in terms of their characterisation and performance is based mainly on measurement methods and procedures, with a lack of emphasis on the need for reliable traceability. Some participants at VAMAS meetings highlighted the need for the authorative approval process for reference materials to be used, although the ISO guides are available for this purpose (see Chapter 7).

In 2004, the VAMAS Steering Committee approved actions aimed at bringing about closer collaboration between VAMAS and CIPM. Seeing the need to widen participation in their activities, VAMAS approached the Director of the BIPM (Bureau International des Poids et Mesures), drawing attention to the need to include materials in the formal international structure for metrology by engaging the attention of NMIs and the CIPM. VAMAS saw this as the best and most appropriate manner to bring international authority and metrological experience to the hugely important and growing area of materials metrology.

Following discussion at the meeting of NMI Directors in September 2004, BIPM hosted a workshop in February 2005 to explore the question, identify specific traceability issues in materials science and propose further international initiatives in the field. The conclusions of this workshop informed a discussion at the 2005 meeting of the CIPM. The CIPM decided to set up an *ad hoc* Working Group with specific Terms of Reference that are given in Section 3.

3 TERMS OF REFERENCE and SCOPE

3.1 TERMS OF REFERENCE

Following the agreement to establish an *ad hoc* Working Group in October 2005, the following Terms of Reference were agreed in January 2006:

- to identify those material properties for which globally comparable, traceable measurement results are important for science, engineering and manufacturing technology;
- to identify those material properties for which the needs for traceable measurements are not covered by the activities of the Consultative Committees; to establish the user needs for activity in materials metrology;
- to investigate the existing capabilities of participating NMIs by initiating some pilot studies, including a small number of interlaboratory comparisons;
- to develop tools and methodologies for establishing traceability in materials testing;
- to define the objectives, aims and initial activities for an ongoing programme in metrology for materials, including recommendations for underpinning activities, such as the organisation of Key Comparisons and the development of Reference Materials and Reference Methods;
- to liaise closely with other interested organisations; and
- to report its conclusions to the 2007 CIPM meeting in November.

3.2 SCOPE

In the course of its deliberations, the Working Group identified a number of different classes of "materials" that would merit consideration under the above Terms of Reference.

Following discussion in the WG, the following categories were agreed to be out of scope for the purposes of this review:

a) liquids (e.g. water, oils, lubricants). OIML had expressed an interest in liquid properties such as viscosity and density, which are important in determining the performance of measuring instruments subject to legal control in some countries (n.b. see below for melts).

b) biological materials (e.g. bone, muscle, organs). Although there is considerable interest in the properties of these materials, for example, in their acoustic properties related to ultrasound scanning or high rate properties for crash modelling analysis, these materials are also considered to be currently out of scope.

Materials in scope are "engineered" materials that are used in design to meet one or, normally, more of structural (e.g. principally mechanical property dependent), other functional (e.g. electrical, optical, magnetic) or decorative purposes. This encompasses materials, such as, ceramics, metals, polymers, concrete and wood. Materials in their molten state, as for example a metal or polymer melt during the processing stage (i.e. casting, injection moulding) or for purposes of reaction, are however considered in scope.

3.3 MATERIAL PROPERTIES – INTRINSIC and PROCEDURAL

Material properties can be divided into those that are considered to be intrinsic (sometimes referred to as inherent or fundamental) and procedural (depending on the characterisation or procedural method used). The NIST Handbook on Materials Metrology [2] usefully characterises these properties in the following tables. Table 3.1 lists as the starting point the SI base quantities and derived units.

Table 3.1 Examples of Measurable Quantities of Conventional Metrology [2]

Category	**Quantities**
Base Quantities	length, mass, time, electrical current, temperature, amount of substance, luminous intensity
Derived Quantities	strain, area, volume, concentration, velocity, acceleration, force, pressure, stress, voltage, charge, capacitance, current density, resistance

Micro-structural properties are given in Table 3.2; intrinsic properties that are independent of test procedure are given in Table 3.3 and procedural controlled properties in Table 3.4.

Intrinsic properties are those that are independent of the measurement procedure and are measurable quantities. In contrast, procedural properties cannot be specified without reference to the test procedure.

Table 3.2 Examples of Measurable Quantities of Structures in Materials Metrology [2]

Category	**Subcategory**	**Quantities**
Unit Structures	Crystallography	lattice parameters, atomic coordinates
	Non-crystalline	radial distribution function
Microstructure	Grains and Pores	size distribution, mean size, aspect ratio, (crystallographic) texture
	Dendrites	mean size, orientation, branch density
	Phase Equilibria	phase transition temperature, phase transition pressure, glass transition temperature, Curie temperature, Néel temperature, triple point temperature
	Interphase Region	thickness
	Magnetic Domains	mean size, aspect ratio, orientation
Surfaces	Topography	asperity mean size, aspect ratio, orientation, surface roughness, crack size, orientation, depth
	Films and Deposits	thickness
Electromagnetic	Charge Distribution	valence, ionicity, covalency, spin, space charge density

Table 3.3 Examples of Measurable Quantities of Inherent Material Properties [2]

Category	Subcategory	Quantities
Mechanical	Elasticity	Young's modulus, shear modulus, bulk modulus, compressibility, Poisson's ratio, elasticity tensor, compliance tensor, sound velocity, Debye temperature
Thermal	Capacity	specific heat, Gruneisen parameter
	Transport	**thermal conductivity, thermal diffusivity, emissivity**, interfacial resistance
	Stability	**thermal expansion**, residual stress
Electromagnetic	Electricity	resistivity, conductivity, thermoelectric power, Hall coefficient, critical current density
	Magnetism	susceptibility, coercivity, critical field strength
	Optics	dielectric strength, index of refraction, permittivity, transmissivity, reflectivity, absorptivity

* *Quantities in bold covered by existing CCs.*

Table 3.4 Examples of Procedural Material Properties [2]

Category	Subcategory	Quantities
Mechanical	Plasticity	ductility, brittle to ductile transition point, **hardness**, creep rate, creep activation energy, creep stress exponent
	Strength	yield strength, proportional limit, tensile strength, flexural strength, shear strength, compressive strength, ultimate strength, fracture toughness, fracture energy, fatigue strength, Weibull characteristic strength
Thermal	Stability	flammability
Durability	Thermomechanical	thermal shock resistance
	Adhesion	adhesive strength
	Tribology	friction coefficient, wear rate, wear coefficient, lubricity, machining rate
Chemical Aging	Corrosion	corrosion rate, activation energy
	Hydration	hydration rate
	Interdiffusion	diffusion rate
Physical Aging	Delamination	delamination rate

* *Quantities in bold covered by existing CCs.*

3.4 EXISTING MATERIALS RELATED WORK IN CIPM

Within the CIPM organisation, the work on SI units is undertaken in Consultative Committees (e.g. CCL = Consultative Committee on Length). The current list of CCs is given in Table 3.5.

Table 3.5 Current Consultative Committees and Materials Activities

	Title	Materials Aspects
CCL	Length	
CCM	Mass and Related Quantities	WGH on Hardness, WGD on Density
CCEM	Electricity and Magnetism	
CCT	Thermometry	WG9 on Thermophysical Properties
CCTF	Time and Frequency	
CCPR	Photometry and Radiometry	
CCQM	Amount of Substance	Several WGs (see text below).
CCRI	Ionising Radiation	
CCAUV	Acoustics, Ultrasound and Vibration	
CCU	Units	

Work on material properties is undertaken currently in the following CCs.

(a) CCM – Working Group on Hardness (WGH).

Hardness measurement is one of the most frequently used test methods since it is fairly simple and easy to conduct. Not only is it convenient, it is a very important material property in practical application. Because of its importance and wide applicability, many of the NMIs in the world maintain hardness standards. Its traceability is very well established by the efforts of the members of WGH and ISO TC164/SC3. It is described systematically in ISO standards so that hardness measurement is a good example to understand the characterisation of materials metrology and how the traceability is established for procedure dependent properties.

For example, ISO standard 6508 for measurement of Rockwell hardness is formed of 3 parts. Part 1 covers the test method, Part 2 covers verification and calibration of the test machine, and Part 3 covers calibration of reference blocks.

The traceability of the results of hardness measurements is transferred via the certified value of CRMs. Certified values of primary hardness block with a declared uncertainty can be obtained through inter-comparison amongst NMIs. This CRM is used to calibrate the hardness machine. Traceability is established through an unbroken chain of comparisons with a stated value and uncertainty of CRM. By using a CRM for calibration of a measurement system, comparability among other accredited laboratories as well as industries can be guaranteed. Although, administered under CCM there is not a dependence on the lowest uncertainty of the kilogram. As noted later there are 16 entries on the CIPM database, formed from 8 key and 8 supplementary comparisons.

Density, a significant material property in all weight sensitive designs, is also covered by CCM, in a working group on density.

(b) CCT – WG9 on Thermophysical properties

WG 9 has an extensive work programme and intercomparisons. Although positioned under the CC for temperature, the work is concerned with thermal properties of materials and these thermal material property measurements do not depend on the highest accuracy of the ITS 90 temperature scales. It is noted that thickness and area measurements are probably the largest contribution to the uncertainty of thermal conductivity. The properties currently under study include thermal expansion (including in CCL for gauge blocks), emissivity and conductivity.

(c) CCQM – several WGs Similar cases can be found in metrology for chemistry, where the result of analysis is affected by the measurement method just as for the procedural properties of materials. For example, CCQM has adopted the Harned cell as the primary method of *pH* measurement and its traceability is established by a CRM, buffer solution, whose value is given by the Harned cell. This is one of the particular features of how the working groups under CCQM establish traceability of amount of substance of similar kind through the hierarchy of measurement method, including the Surface Analysis Working Groups (listed below), in which measurands are closely related to material properties.

- CCQM Working Group on Gas Analysis
- CCQM Working Group on Electrochemical Analysis
- CCQM Working Group on Inorganic Analysis
- CCQM Working Group on Organic Analysis
- CCQM Working Group on Bio-analysis
- CCQM Working Group on Surface Analysis

(d) CCEM – no materials WGs

There are no materials working groups, but measurements are undertaken at NMIs for both dielectric and magnetic properties of materials. Low permeability reference blocks for magnetic measurements are produced that are based on iron-filling loaded acrylic blocks. It is noted that solid metals are also used as reference materials. Dielectric references are normally based on liquids, even when a solid material would be more appropriate. This in part due to the lack of long-term stability of sold polymeric reference material. In a similar manner to thermophysical properties, thickness measurements are a critical factor in these measurements.

(e) CCAUV – no WGs

There are no working groups, but measurement of acoustic material properties are routinely undertaken. For example, a filled gel-like material is used to check the heating effect of ultrasound used in medical applications. In addition, the speed of sound in a material is a well-established property. Other properties measured include the attenuation (or transmission/insertion loss), which is particularly relevant to stealth and other applications where high absorption is needed. In addition, the proportion of attenuation due to scattering and absorption is of interest.

> **Comment: Material properties are already covered in several CCs. including in some cases through dedicated working groups. It is clear that there are opportunities for other materials working groups. Their inclusion in the CC does not require the highest precision of the corresponding SI units (e.g. dielectric or thermal conductivity), but should contribute to establish improved traceability and precision.**

4 TRENDS IN METROLOGY – BIPM/CIPM, MRA and EURAMET

4.1 TRENDS IN METROLOGY

While the Metre Convention, originally established to maintain the metre and the kilogram, has overseen the development of the complete SI system of units, it has also responded to the changing needs for quality control of measurements in new industries and new technologies. For example, new work has been initiated in the areas of chemistry, medicine and pharmacology.

An updated 3rd version of the report on *"evolving needs in metrology"* [3] produced in 2007 notes in the foreword that the demand for traceability has increased faster than expected and in new areas; and that the needs cover all areas of human endeavour, as noted below,

> "Over the last four years, it has become clear that there are additional needs for internationally reliable, recognised and comparable measurement results and traceable to long term stable measurement standards. Therefore, the CIPM is pleased to submit an updated *Report on the Evolving Needs for Metrology in Trade, Industry and Society and the Role of the BIPM* to the Governments of the Member States."

and that,

> "The demand for reliable, accurate and comparable measurements and test results is still growing rapidly in industrial production, in trade or in society, and in areas such as food safety, health care, environmental studies, forensic science and security."

Under the heading of "improving the quality of measurement standards" it is noted that,

> - "The demand for traceable measurements of the properties of materials is growing in many areas, for example, in electromagnetic, thermo-electric, thermodynamic, optical and mechanical properties. In addition, the interest in properties of fluids and gases and structural properties is growing rapidly. The development of new advanced materials (ceramics, polymers, carbon-strengthened materials) and their application in health care, aeronautics and space industries, and the construction industry are the driving force for more accurate, traceable measurements."

and that the needs are driven by

> - "Accreditation agreements for calibration, measurement and testing laboratories and inspection bodies are only credible when measurement results and measurement uncertainty statements are comparable and traceable to the same references. Likewise, product certification and other conformity assessment statements require traceable measurements, carried out with regularly calibrated measuring devices."

The report correctly anticipates the conclusion of this current report that materials properties are an important and yet unexplored area of measurement science, as follows:

3.4 "Measurement standards in the field of material properties

Industry has recently expressed an increased demand for traceable measurements of materials properties. In particular, organizations like the Versailles project on Advanced Materials and Standards (VAMAS) and the APEC Network for Materials Evaluation Technology (ANMET) and a number of NMIs with activities in this field are coming together to address the importance of traceable measurements.

This field encompasses a wide field of material properties, such as:

- electromagnetic properties (magnetic and dielectric),
- thermo-electric properties,
- thermodynamic/thermophysical properties (conductivity, heat transfer, phase analysis, expansion, heat capacity, emissivity, diffusivity),
- optical properties,
- mechanical properties (hardness, modulus, strength, toughness, fatigue, creep, friction, corrosion, lubrication),
- properties of fluids and gases (viscosity, density, calorific value), and
- structural properties (composites, aerosols, gels, grain and particle size distribution, porosity, defects, shape).

As the measurement characteristics of many materials may emerge from industrial-specific requirements, the subjects needs to be addressed broadly so as to identify the metrological needs and the related support which may be needed from the NMIs in addition to the standardization carried out by the industry sector itself. With this in mind, the CIPM has created an *Ad-hoc* Working Group on Materials Metrology which should report back by the end of 2007 on any actions needed by the NMIs in support of the establishment of appropriate internationally recognized, comparable and traceable measurements and test results"

In addition, several of the examples of the needs in nanotechnology are materials based, as shown below.

3.3 Nanometrolgy

The activities indicated under the heading nanometrolgy cover a wide range of disciplines, including dimensional measurements, electromagnetic measurements, sub-micro electromechanical devices and sub-nano vibration devices, optical microscopy, chemical analysis, surface analysis, structural material property analysis, bio-technology and micro-biology. Nano-technology is rapidly becoming a multi-disciplinary subject.

Current challenges which are being addressed include:

- manufacturing at the micro- and nano-scale (e.g. nano-lithography as used in the semiconductor industry),
- nano-structured materials (e.g. carbon nano-tubes),
- new composite materials (manipulation of nano-scale particles from various materials),
- nano-electronics, nano-photonics, nano-magnetics (e.g. molecular electronics).
- energy conversion and storage (e.g. nano-rod polymer solar cells and flexible sheets),
- nano-scale instrumentation (e.g. chip based and single electron devices),
- computer circuits (e.g. to construct energy efficient circuits of molecules and atoms),
- energy saving (e.g. the development of new energy sources like hydrogen fuel),
- advanced chemical, biological, radiation and explosive detection (e.g. chip for detecting PSA and drug discovery),
- healthcare, therapeutics and diagnostics (e.g. peptide nano-tubes as antibiotics, puncturing the cell wall and gold nano-particles to deliver DNA molecules safely into cancer cells),
- the cosmetics industry,
- DNA and RNA strands (e.g. observing and manipulating strands in order to understand fundamental problems in biology), and
- nano-scale processes for environmental improvement (e.g. nano-particle water filtration)."

4.2 JOINT COMMITTEES AND COOPERATION AGREEMENTS

With time, metrology needs have emerged in new areas. It is for instance noted on the CIPM web-site that *"The goal of obtaining comparability of laboratory diagnostic test results will be possible only when common reference systems can be established for worldwide use. A critical step in reaching this goal is achieving traceability of reference measurement procedures and reference materials to a universally recognised and accepted reference point such as the International System of Units (SI). Recently, traceability requirements for medical devices marketed within the European Community have been codified. The European Community In Vitro Diagnostic Directive states that "**The traceability of values assigned to calibrators and/or control materials must be assured through available reference measurement procedures and/or available reference materials of a higher order.**" (98/79/EC, Annex 1 (A) (3) 2nd paragraph).*"

Therefore, the International Bureau of Weights and Measures (BIPM), the International Federation for Clinical Chemistry and Laboratory Medicine (IFCC), and the International Laboratory Accreditation Cooperation (ILAC) have established the Joint Committee for Traceability in Laboratory Medicine (JCTLM) to provide a worldwide platform to promote and give guidance on internationally recognised and accepted equivalence of measurements in Laboratory Medicine and traceability to appropriate measurement standards. The principles are embodied in ISO 17511 and 18153.

More recently, BIPM and International Commission on Illumination have signed a cooperation agreement. The Agreement recognises the responsibilities and roles of the BIPM and the CIE and emphasises the need to consult together to ensure that data related to measurements of light, optical radiation, colour, optical properties of materials, and photobiological and photochemical quantities are kinds-of-quantity (ref. section 1.2 of ISO Guide 99) traceable to the International System of Units (SI).

The Agreement also recognises the importance of the international recognition and acceptance of measurement procedures for these quantities, which can be provided through the Mutual Recognition Arrangement of the International Committee for Weights and Measures, the CIPM MRA. Both parties to the Agreement will be represented at meetings organised by the other party, especially those which relate to the liaison needed as a result of the CIPM's responsibility for the definition of the photometric units in the SI and the CIE's responsibility for the standardisation of the action spectra of the human eye.

Other new needs foreseen by Wallard [4] include "soft" metrology related to the touch, sound and smell of a product, the development of improved techniques for making real-time measurements and increasing demands for the measurement of ever smaller or faster events. Reference is also made in this paper to the area of materials metrology and the work of the current ad-hoc working group.

Comment: There is general recognition and acceptance of widening coverage by CIPM of measurement areas outside the initial SI responsibilities.

4.3 MRA - MUTUAL RECOGNITION ARRANGEMENT

The mutual recognition arrangement (MRA) drawn up in 1999 by the CIPM seeks to provide "a secure technical foundation for wider agreement for measurements related to international trade, commerce and regulatory affairs". This is normally achieved through international comparisons of measurements - "key comparisons" - undertaken by those laboratories working at the highest level and organised by the CIPM's Consultative Committees (CCs) or through key and supplementary comparisons organised by regional metrology organisations.

These comparisons were recognised as being different from earlier CC comparisons, which were usually performed with the aim of establishing the realisation of SI units and their associated uncertainty. In the MRA comparisons each participant carries out the measurements without knowing the results of others until the comparisons have been completed. They provide an independent assessment of performance and are similar to the round-robin exercises carried out by VAMAS over many years, or in more limited cases by Technical Committees within ISO (International Organisation for Standardization). The participants also need to demonstrate that a quality system has been installed, which meets the requirements of ISO 17025 or a similar standard. Through these two components of demonstrated technical equivalence and assessed quality systems, the MRA process provides confidence in the validity of calibration and measurement certificates issued by all the participating laboratories.

Comment: The need for, and benefits of, mutual recognition applies equally strongly to material property determination to meet trade and regulatory requirements.

On the basis of these comparisons, the participating laboratories have published on the BIPM website lists of their Calibration and Measurement Capabilities (CMCs) for a wide range of calibration and measurement services. Although the vast majority of these CMCs, which have been validated by the other national laboratories, refer to the calibration of standards, there are, for instance, entries for material properties covering hardness, surface analysis, thermal and electrical conductivity properties.

Comment: A range of material properties is already covered by key and supplementary comparisons.

A similar approach was adopted by NPL in developing a standard qualification plan (SQP) for polymer matrix composites properties, whereby preliminary design data will be released by the material supplier through using agreed procedures. As part of the validation of this approach round-robin assessments of the capability of all participating laboratories to produce reliable data were undertaken [5]. The SQP is a more developed version of the database standards, such as ISO 10350 [6], aimed at ensuring comparable data for plastics. This is achieved by selecting specific options in test method standards, when options are included in the standard, to ensure comparable data are obtained.

4.4 EURAMET – iMERA MATERIALS ROAD MAP

Under the EURAMET programme, a recent initiative has been developed to provide a unified approach to metrology and collaborative projects under the MERA (Metrology in the European Research Area) programme. As part of a second implementation phase (iMERA) road-mapping was undertaken for the established SI units and Euramet committees. In addition, several new measurement areas were also reviewed for their relevance to meeting emerging societal needs. These new areas were biosciences, modelling and materials, with the last area convened by NPL.

As new materials will impact on all aspects of industrial, infrastructure and quality of life activities, this road-map focuses on three, of several, aspects of potentially high impact on the major societal requirements over a 10-20 year timescale. The areas chosen were:

- *I.* *Maintaining full human personal activity,*
- *II.* *Increasing European competitiveness through "designed for function" materials,*
- III. *More effective energy conversion, storage and utilisation.*

The specific opportunities in the selected areas were,

I. Maintaining full human personal activity – demand from the ageing society, but also from people with injuries or birth deformities, to increase their quality of life through implants, both passive (e.g. hip joints) and active (e.g. hearts), including tissue scaffolds. It is important to determine lifetime performance, as people now outlast implants (i.e. implants are being installed in more active people who have a longer life expectancy). Surface measurements are relevant to wear and cell compatibility issues. There is a need to optimise and validate implant performance to reduce both patient trauma from a repeat operation and hospitalisation costs.

II. More effective energy conversion, storage and utilisation – the EU energy agenda requires a two-pronged approach of better conservation, including reduced storage and transmission losses, and new conversion processes. Improved **energy conservation** is needed in construction and non-ambient processes, such as engines, process plant etc. Major quantities of energy are available from sunlight through improved photovoltaics – the roof tile of the future - with minimum environmental negative impact. There is a need to support improved PV technologies through materials characterisation and measurements to understand and improve lifetime efficiency issues to maintain the "tile" efficiency.

III. Increasing European competitiveness through "designed for function" materials – many areas of industrial activity and export opportunities require optimised properties related to the specified needs, including metamaterials, graded or hybrid structures whose added value can give unique solutions. Especially new micro- and nano-optical structures, e.g. photonic crystals as well as metamaterials (and combinations of both), will revolutionise information and communication technologies as well as imaging technologies and the related metrology. This may include optimising performance to allow efficient recycling, demonstrating a full "life-cycle " approach.

The measurement issues supporting these requirements were:

A. Physical-chemical property measurements- **measurement of electro-chemical processes, including at the nano-scale,** to provide thermodynamic input into fuel cell characterisation, and ageing processing. (Applies to example II mainly).

B. Structural analysis techniques - **absolute mapping of material structure, including anisotropy and 3D** using techniques such as X-ray tomography, confocal microscopy and 3D electron microscopy characterisation, (Applies to example I, II and III).

C. Constitutive property measurements. – there is a need to provide **early detection of damage and to quantify ageing** at the micro-structural level in order to assess residual life in infrastructural plant, implants etc, and as an aid to material development. Techniques such as nanoindentation need development, validation and traceability to SI units or reference material as appropriate. **High strain rate** data and test methods are specific requirements for transport crash modelling . (Applies to example I, II and III)

D. Validated materials modelling and data – validation of the **prediction of Gibb's constant** from ab-initio calculations would enable faster material developments tailored to specific applications; identification of the **sensitivity of predictive models** to input data variability can drive a fitness-for-purpose approach to measurement accuracy and uncertainty. (Applies to example I, II and III)

The primary measurement requirement is to demonstrate robustness in use with the required precision and traceability to SI units or similar references, either for different, often competing material classes, or can all be used to provide comparative information (e.g. dielectric, ultrasonic, spectroscopic and thermal measurements of cure). The measurement techniques needing development can be related to the underpinning materials measurement research categories in one of the participating NMIs (NPL) - see Table 4.1.

Table 4.1 NPL Materials Measurement Infrastructure

Area	Individual technique/tools area	Calibration/traceability
A. **Phys-Chem properties of materials**	1. Thermodynamic data (Gibb's energy, wettability, polarisability)	Reference materials,
	2. Thermal analysis (Tg, reaction energies from DSC, DMA, PVT measurements)	Temperature, Force/mass, Displacement / length
	3. Electrochemistry, (link with thermodynamics data, pH, mass transport, capacitance)	voltage, current, standard reference electrode
B. **Structural analysis of materials**	1. Microstructure and topological characterisation (Optical, scanning, EDAX, WDX, EBSD, AFM)	Reference images/artefacts Microscope grids
	2. Compositional information (Spectroscopic chemical analysis/surface analysis)	Reference Materials
	3. Non-destructive evaluations (Ultrasonics, X-ray, EPSI, thermography, Acoustic Emission)	Reference images,
C. **Properties of materials**	1. Fluid flow (Rheology, surface tension, wetting, density)	Force / mass, volume Displacement / length
	2. Non-mechanical testing (electrical, dielectric, magnetic, optical, thermal expansion/conductivity, heat transfer)	current. voltage, length etc.
	3 Mechanical testing (Non-contact strain, strength/modulus at different scale, nano-indenter)	Force / mass Displacement / length
D. **Modelling of materials**	1. Predictive modelling (thermodynamic, micromechanics, molecular modelling)	Reference data
	2. Analytical and numerical modelling (FEA, CFD, CoDA) Model testing/comparison/validation	Reference constitutive equations and parameters.
	3. Uncertainty budgeting	Uncertainties on constants/data

4.5 VERSAILLES PROJECT ON ADVANCED MATERIALS AND STANDARDS

The Versailles Project on Advanced Materials and Standards was established in 1982 following a G7 economic summit held in Versailles. Subsequently, a Memorandum of Understanding was signed by senior representatives of government in countries of the Economic Summit (G7) and of the European Community. The main objective of VAMAS is to support trade through international collaborative projects aimed at providing the technical basis for harmonised measurements, testing, specifications, and standards in order to increase the take-up of advanced materials. The scope of such collaboration embraces many aspects of measurement, including the development of test methods, participation in intercomparisons, production of reference materials and materials databases, and establishing agreement on nomenclature: all items that are often required as a precursor to the drafting of standards.

Through its activity, VAMAS fosters the development and harmonisation of international standards for advanced materials by the various existing standards agencies. The scope of this international collaboration embraces all aspects of enabling science and technology required as a precursor to the drafting of standards. The specification of materials in terms of their characterisation and their performance is based mainly on measurement methods and procedures, without particular emphasis on the need for reliable traceability.

VAMAS conducts its work through Technical Working Areas. Thirty-one TWAs have been established, with 12 having been terminated following completion of their work. VAMAS has contributed to at least 77 standards, including 36 ISO standard, 9 IEC, 13 CEN and 12 ASTM standards [7]. In addition, ISO/IEC have published 5 TTAs (Technical Trends Assessments) through the liaison agreement. Participation has been mainly from G7 countries, with 627 participants, and 111 participants from other countries.

Current work areas include:

TWA 2	Surface Chemical Analysis.
TWA 3	Ceramics for Structural Applications
TWA 5	Polymer Composites
TWA 16	Superconducting Materials
TWA 17	Cryogenic Structural Materials
TWA 18	Statistical Techniques
TWA 20	Residual Stress
TWA 22	Mechanical Properties of Films and Coatings
TWA 24	Performance Related Properties of Electroceramics
TWA 26	Full Field Optical Stress and Strain Measurements
TWA 27	Ceramic Powder & Green Bodies.
TWA 28	Spectrometry of Synthetic Polymers
TWA 29	Nanomechanics Applied to Scanning Probe Microscopy
TWA 30	Tissue Engineering
TWA 31	Creep, Crack and Fatigue Growth in Weldments
TWA 32	Modulus Measurement
TWA 33	Polymer Nanocomposites
TWA 34	Nanoparticle Populations

Recently, a new MoU was agreed by the founding members that will broaden the global coverage of the VAMAS organisation by making it possible for non-G7 countries to join the Steering Committee. Several countries are already involved in the current CIPM *ad-hoc* Working Group or have participated in technical work. Countries planning to join are Australia, Brazil, Chinese Taipei, India, Korea, Mexico and South Africa.

VAMAS activities are based frequently on round-robins to establish, firstly, reliable methods fit-for-purpose and secondly, to determine precision data encompassing the repeatability and reproducibility of the preferred method. The finalised and validated methods are then offered to the standards development organisations, such as ISO, ASTM, CEN, simultaneously on behalf of all participants as the preferred optimised solution to meet the identified need. VAMAS is one of the few organisations worldwide providing precision data supporting draft standards.

For example, in figure 4.1 the results of a round-robin for mode II fracture energies for a carbon and a glass-fibre reinforced polymer composite using two different starter crack conditions. This work was undertaken in TWA 5. In TWA 22 reference materials are being developed for micro-indentation.

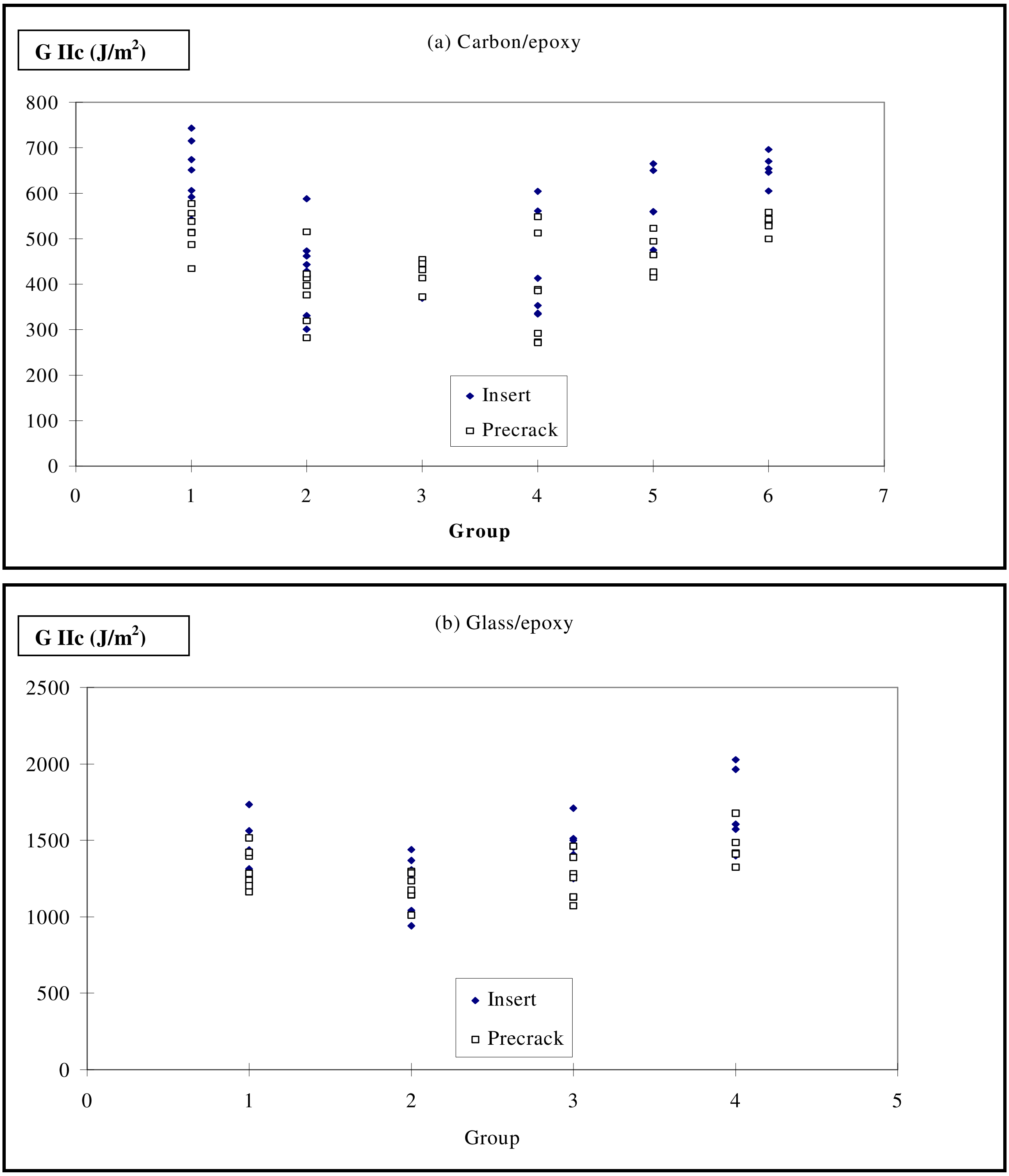


Figure 4.1 Initiation fracture energy, G IIc (J/m^2) based on non-linear response point [8]

At a special seminar hosted at LNE to celebrate the 25th Anniversary of the creation of VAMAS, speakers highlighted the extensive future challenges for measurements in the fields of bio- and nano-materials.

5 VIM AND MATERIALS METROLOGY DEFINITIONS

5.1 VIM (INTERNATIONAL VOCABULARY OF METROLOGY) DEFINITIONS

The VIM definitions have recently been revised [9], as for example for the “quantity” term given in Table 5.1.

Table 5.1 Definition of quantity from VIM - ISO Guide 99 [9]

1.1

quantity
property of a phenomenon, body, or substance, to which a number can be assigned with respect to a reference

EXAMPLE

Example of quantity in a general sense		**Example of particular quantity**
length, *l*	radius, *r*	radius of circle A, *r*A or *r*(A)
	wavelength, λ	wavelength of the sodium D radiation, λ D or λ (D; Na)
energy, *E*	kinetic energy,	*T* kinetic energy of particle *i* in a given system, *Ti*
	heat, *Q*	heat of vaporization of sample *i* of water, *Qi*
electric charge, *Q*		electric charge of the proton, *e*
electric resistance, *R*		electric resistance of resistor *i* in a given circuit, *Ri*
amount-of-substance concentration of entity B, *c*B		amount-of-substance concentration of ethanol in wine sample *i*, *ci*(C2H5OH)
number concentration of entity B,*C*B		number concentration of erythrocytes in blood sample *i*, *C*(Erys; B*i*)
Rockwell C hardness (150 kg load), HRC(150 kg)		Rockwell C hardness of steel sample *i*, HRC*i*(150 kg)

NOTES
1 — In English, the term “quantity” is often used for **kind of quantity**. In French, the term “nature” is only used in expressions such as “grandeurs de même nature” (in English, “quantities of the same kind”).
2 — A reference can be a **measurement unit**, a **measurement procedure**, or a **reference material**.
3 — Symbols for quantities are given in the International Standard ISO/IEC 80000, Quantities and units. The symbols for quantities are written in italics.
4 — The preferred IUPAC/IFCC format for designations of quantities in laboratory medicine is “System—Component; kind-of-quantity”.
Example: “Plasma (Blood) Sodium ion; amount-of-substance concentration equal to 143 mmol/l in a given person at a given time”.
5 — A quantity as defined here is a scalar. However, a vector or a tensor whose components are quantities is also considered to be a quantity.
6 — In case of ambiguity, the term ’quantity’ may be qualified, e.g. ‘physical quantity’.

It is noted that the VIM examples of “quantity”, see figure 5.1, includes the procedural material property “hardness”.

The VIM defines **metrology** as “field of knowledge concerned with measurement”. It is noted that “Metrology includes all theoretical and practical aspects of **measurement**, whatever the measurement uncertainty and field of application”.

The VIM introduction notes that *“In this Vocabulary, it is taken for granted that there is no fundamental difference in the basic principles of measurement, whether the measurements are made in physics, chemistry, laboratory medicine, biology, or engineering. Furthermore, an attempt has been made to meet conceptual needs of measurements in fields such as biochemistry, food science, forensic science, and molecular biology”*. There is no reason why these comments do not also apply to measurements in the field of materials.

Metrological traceability is defined as *"property of a **measurement result** whereby the result can be related to a stated reference through a documented unbroken chain of **calibrations,** each contributing to the **measurement uncertainty",*** where for this definition, note 1 states that: "*A 'stated reference' can be a definition of a **measurement unit** through its practical realisation, or a **measurement procedure** including the measurement unit for a **non-ordinal quantity,** or a **measurement standard"**.*

It should be noted that even when traceability is established to a "stated reference", "*metrological traceability by itself does not ensure adequate measurement uncertainty or absence of mistakes* (see Note 5 of this definition)".

Definitions for several other selected terms are summarised in Table 5.2. Reference [9] should be consulted for the full set of terms and the associated notes (c.f. notes in Table 5.1).

Table 5.2 Summary of selected definitions from VIM - ISO Guide 99 [9]

Clause	Term	Description
1.1	**quantity**	property of a phenomenon, body, or substance, to which a number can be assigned with respect to a reference
1.5	**derived quantity**	**quantity**, in a **system of quantities**, defined in terms of its **base quantities**
1.9	**measurement unit** **unit of measurement unit**	scalar **quantity**, defined and adopted by convention, with which any other quantity of the same **kind** can be compared to express the ratio of the two quantities as a number
2.3	**measurand**	**quantity** intended to be measured
2.9	**measurement result** **result of measurement**	set of **quantity values** being attributed to a **measurand** together with any other available relevant information
2.10	**measured quantity value** **measured value of a quantity** **measured value**	**quantity value** representing a **measurement result**
2.27	**measurement uncertainty** **uncertainty of measurement** **uncertainty**	parameter characterising the dispersion of the quantity values being attributed to a measurand, based on the information used.
2.39	**calibration**	operation that, under specified conditions, in a first step establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication.
2.41	**metrological traceability**	property of a measurement result whereby the result can be related to a stated reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty.
2.42.	**metrological traceability chain** **traceability chain**	sequence of measurement standards and calibrations that is used to relate a measurement result to a stated reference
2.43	**metrological traceability to a measurement unit** **metrological traceability to a unit**	metrological traceability where the stated reference is the definition of a measurement unit through its practical realisation
2.46	**metrological comparability of measurement results** **metrological comparability**	comparability of measurement results that are metrologically traceable to the same reference

NOTE: The expression 'traceability to the SI' means metrological traceability to a measurement unit of the International System of Units.

Comment: - the re-definition allows wider options for traceability such as that used by the JCTLM in assessing reference materials.

5.2 DEFINITION of MATERIALS METROLOGY

5.2.1 NPL / VAMAS PROPOSAL

The initial definition proposed by NPL and adopted by VAMAS in 2004 was:

Materials Metrology: "Development and application of traceable measurements and predictive modelling to the structure, properties and performance of materials throughout their life cycle enabling their efficient use", where *Materials Metrology* is taken to mean the "metrology applied to the measurement of material properties" (includes microstructural characterisation).

5.2.2 NIST PROPOSALS

Alternative definitions due to NIST publication SP960-11 [2] are given in figure 5.2.

- *Material*: An assemblage of matter delimited in space.
- *Materials Metrology:* The science of measurement applied to the characterisation, understanding, and development of materials.
- *Material Property*: A quantifiable response of a material to an externally applied stimulus.
- *(Measurable) Quantity*: [VIM 1.2] An attribute of a phenomenon, body, or substance that may be distinguished qualitatively and determined quantitatively.
- *Procedural Quantity*: A quantity whose identity is determined as an attribute of a phenomenon, body, or substance and an attribute of a measurement procedure, and that may be distinguished qualitatively and determined quantitatively.
- *Inherent Material Property*: A material property that is a measurable quantity.
- *Procedural Material Property*: A material property that is a procedural quantity.

Figure 5.2 Definitions from SP960-11 "Data Evaluation Theory and Practice" [2]

5.2.3 SCOPE OF THE AD-HOC WORKING GROUP (WGMM)

The WG agreed to scope its materials metrology activities as follows.

Materials Metrology covers the application of measurement knowledge to the determination of the intrinsic and procedural properties of materials, including compositional and micro-structural properties.

Where compositional properties = elemental data (CCQM), micro-structural properties = Table 3.2, intrinsic properties = Table 3.3 and procedural properties = Table 3.4.

6. THE IMPORTANCE OF THE ACCURATE MEASUREMENT OF MATERIAL PROPERTIES

Government bodies and regulators are constantly called upon to make decisions related to:

- Protecting the health and welfare of consumers and the public
- Protecting the environment
- Developing new regulations and requirements
- Assessing compliance with regulatory and legal requirements
- Allocating resources, both technical and financial

For companies, traceability of measuring and test equipment to national standards by means of calibration is necessitated by the growing national and international demand that manufactured parts be interchangeable: supplier firms that make products and customers which install them with other parts must measure with the "same measure". But there are legal as well as technical reasons for ensuring equality of the measurements. The relevant laws and regulations have to be complied with just as much as the contractual provisions agreed with the purchaser of the product (guarantee of product quality) and the obligations to put into circulation only products whose safety is not affected by defects if they are used properly.

Equally manufacturers must demonstrate traceability of material data used in design, for qualification and for manufacture. Some examples of the infrastructure required to meet these needs are described in this section.

6.1 SUPPORT TO REGULATON

Materials support and underpin all human activity, and both the materials and their ultimate products are increasingly supplied on a global scale. In the UK alone, the material supply and direct fabrication industries are worth £200 billion and forms 15% of the GDP. In all these cases measurements are undertaken to ensure "fitness for purpose".

Several examples are given in the following text.

Case study 1 – Bolted steel construction

To meet the European Construction Products Directive for a bolted steel construction, adherence would be made to the Eurocode Part 3 on design of steel structures. In turn this document calls up ISO 898, for the bolts used in the construction. The quality of the bolts is controlled in this standard by a comprehensive set of material properties (i.e. the tensile strength, lower yield stress, proof stress, impact strength, Brinell, Rockwell and surface hardness, elongation after fracture). Each of these properties is measured according to an appropriate test method, such as, ISO 6892 for tensile properties.

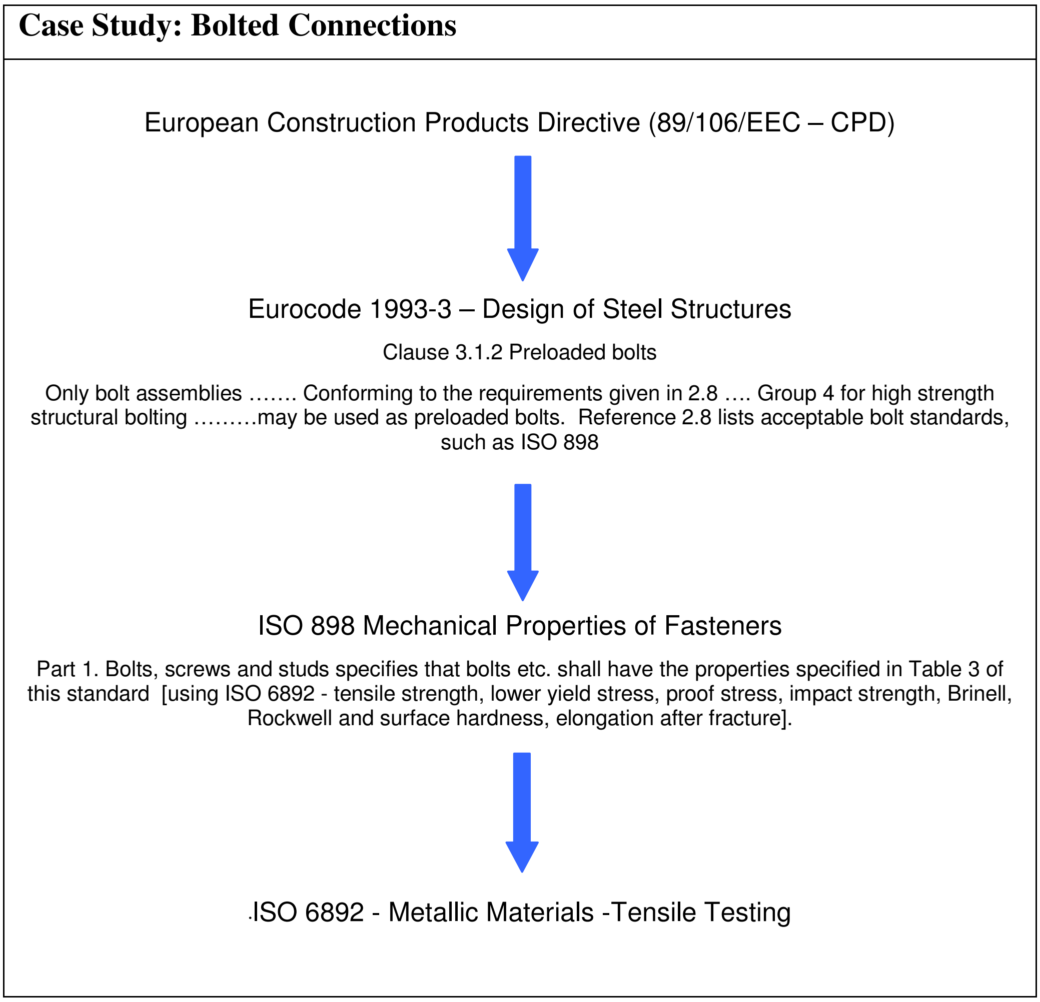


Figure 6.1 Traceability to material properties for bolted connections in steel construction.

Case study 2 – Gas cylinder approval

A second example is given in the area of gas cylinders as used in breathing kits for fire rescue staff, which is similarly driven by a European Directive. In this case, there is even a long-term chemical environment test requirement.

In the UK, NPL was authorised by the Health and Safety Executive to undertake part of this certification. Cylinder approval was by obtained through meeting required levels related to ultimate tensile properties, intergranular corrosion and impact performance.

Case Study – Gas Cylinders

Conformity assessment for UN certified pressure vessels for transport of dangerous goods

↓

European Pressure Vessel Directive

↓

ISO 18172 – Refillable welded stainless steel cylinders

- controlled minimal material properties include tensile strength, impact strength and intergranular corrosion
- Also product tests such as burst strength
- EN 15047 controls the minimum tensile elongation and impact strength

↓

ISO 6892 - Metallic Materials -Tensile Testing

ISO 3651- Determination of intergranular corrosion

EN 10002 Tensile/EN 10045 Impact

Figure 6.2 Traceability to material properties for liquid gas cylinders.

Case study 3 – F1 Racing car - material approval

A third example, although easy to trivialise, with serious economic and safety impact, concerns Formula 1 racing, a multi-million dollar industry. In this case, some materials that can be used within the racing car are limited to a specific modulus value that must not be exceeded. The racing car industry was experiencing difficulty with measuring this modulus and obtaining agreement, to the extent that although a team may measure the material as acceptable, the scrutinisers would not. This inconsistency is not surprising in view of the comparative results from round-robins discussed later in section 14. NPL was already working independently with more than one F1 manufacturer to measure more consistent data, when approached by the FIA to establish a mandated facility that all teams used. A method was developed by NPL, together with the FIA and made available to the sector through NPL.

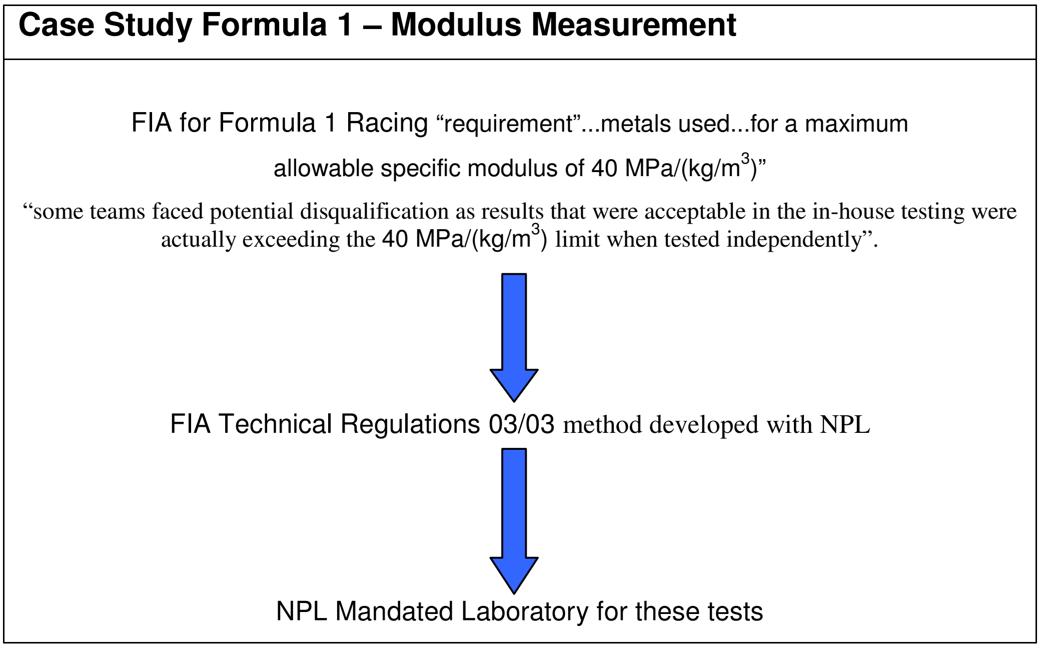


Figure 6.3 Traceability to modulus properties for racing car components

6.2 SUPPORT TO TEST LABORATORY ACCREDITATION

Government bodies and regulators must have confidence in the data generated by test laboratories in order to make decisions. Using an accredited laboratory can help establish and assure this confidence. If a reputable accrediting body, such as the signatories of the ILAC agreement, accredits a laboratory it means that the laboratory has achieved a prescribed level of technical competence to perform specific types of testing, measurement and calibration activities. The result is assurance that the laboratory is capable of producing data that are accurate, traceable and reproducible - critical components in governmental decision-making.

In many instances, laboratory accreditation relies on the availability of certified reference materials as noted in the introduction of ILAC-G12: 2000 [Guidelines for the Requirements for the Competence of Reference Materials Producers] that " One of the key factors affecting laboratories' capabilities to produce reliable test data is the availability of reference materials with property values that can be relied upon by their users". There is also a necessity to have agreed methods or procedures, as provided through international standards or otherwise agreed.

This aspect is discussed further in the following section.

6.3 SUPPORT TO DESIGN, QUALITY CONTROL AND MATERIALS DEVELOPMENT

Extensive testing is undertaken continuously on a significant scale in support of materials development and supply, qualification, design and manufacture.

A recent survey in the UK [10] investigated the need for material property data on engineering materials. The survey covered all material types, all applications and industrial sectors, and all sizes of companies. In this report only a selection of the information obtained is reported. In figure 6.4 is shown the degree of uncertainty needed for all replies at different stages of the design and manufacturing process. As noted by comparison with data given elsewhere in this report, the user required level of uncertainty is challenging.

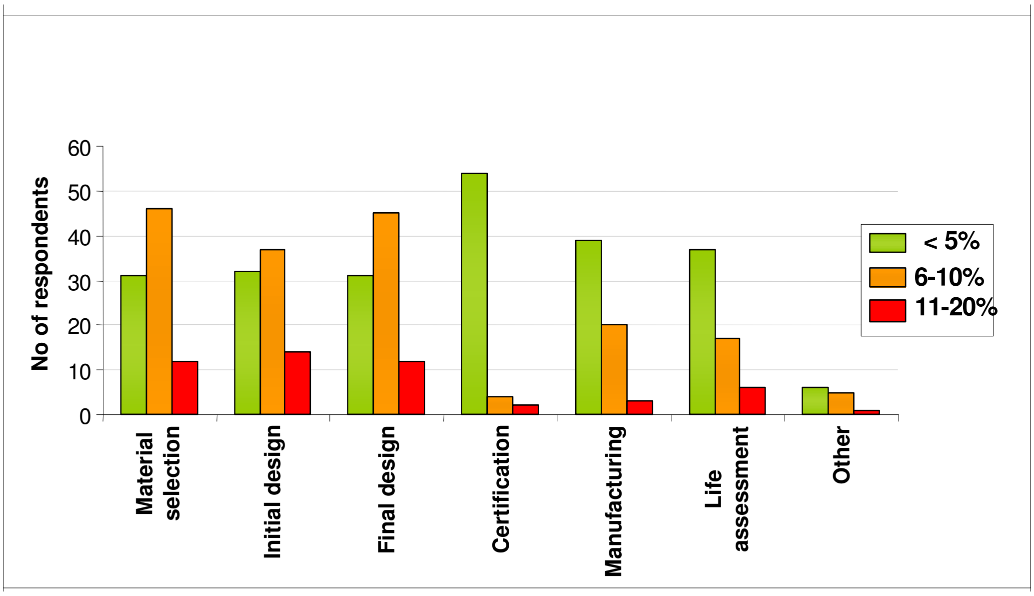


Figure 6.4 User required precision of material properties at different stages

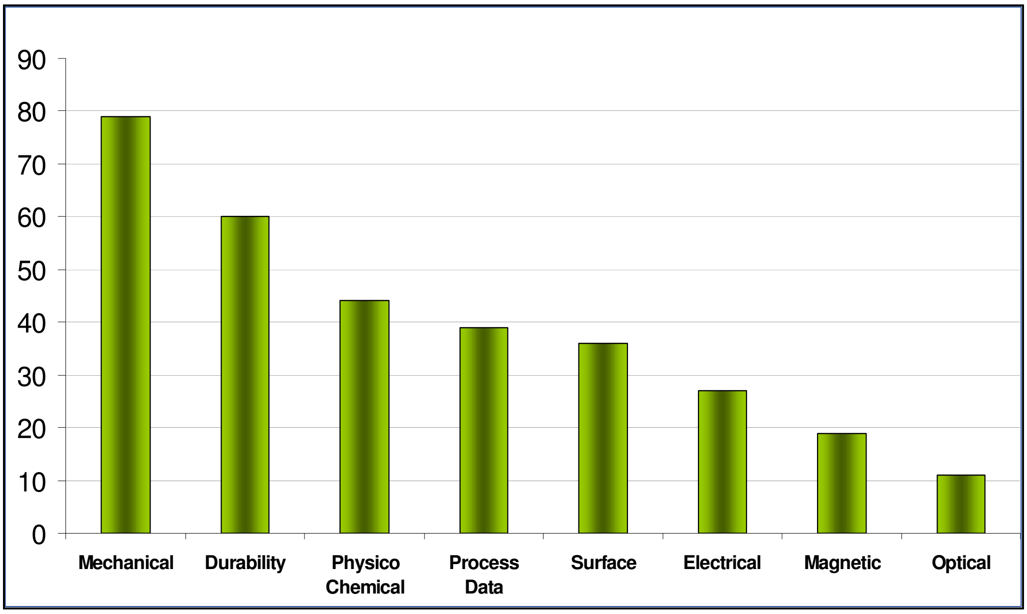
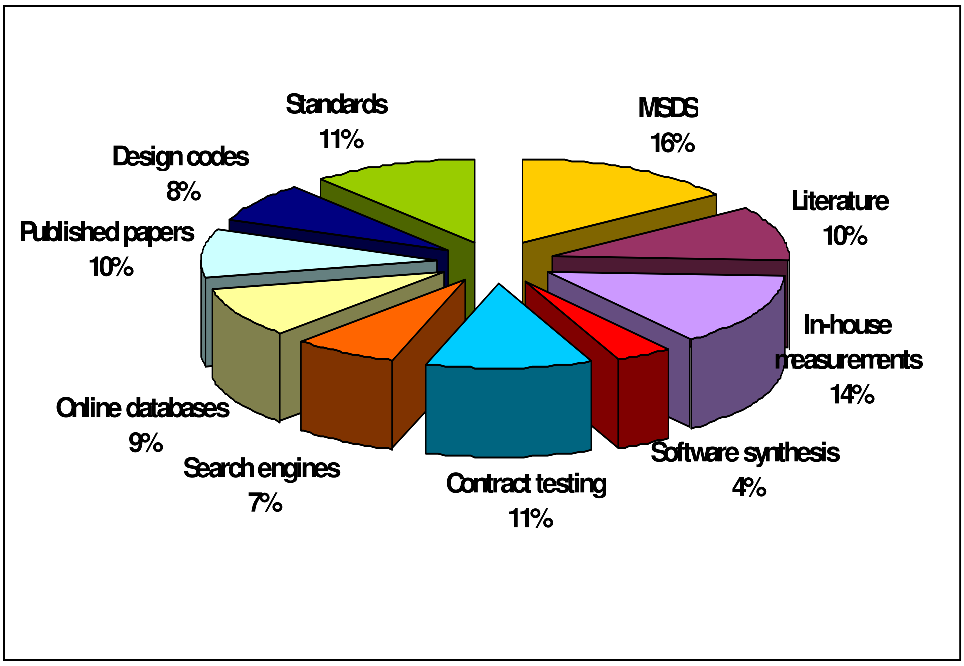


Figure 6.5 Range of outstanding property requirements

The correspondents were also asked to indicate where they had unsatisfied requirements. As indicated in Figure 6.5, this covered a full range of properties (in contrast to the often over-emphasis on mechanical properties only).



** MSDS = Material Suppliers Data Sheets*

Figure 6.6 User sources of materials data.

Finally it is noted that the sources of data are numerous and varied, as shown in Figure 6.6. It is important, in future, that the level of confidence in any data is known, with a continuing drive to ensure that the trend is to obtain lower uncertainty.

Comment: The survey found that users have high expectations for the accuracy of their design data for a wide range of properties. Generally, for all phases of the design, manufacture and in-service life application, data was required with better than 10% accuracy and preferably better than 5%. These requirements are in advance of currently available precision. As data is sourced from a wide range of sources, it is necessary to ensure that all these routes use best metrology practice.

6.4 ECONOMIC IMPACT

Accurate measurement of material properties can have a considerable direct economic cost in the end application. For example, economic loss due to accidents and defects of industrial products is estimated in Japan to be as high as 4% of GDP. Prevention of those accidents is important not only to secure safety of people, but also to support economic growth. Major reasons of those accidents are generally defined as a result of insufficient quality data in fatigue, corrosion and abrasion, and creep of materials. For those high temperature structural parts, which are used for power-generation plants, petrochemical plants, and jet planes and automobiles, creep defects are one of major reasons of material degradation.

For power-generation plants in Japan, cost savings of approximately 100 million yen/day can be obtained based on creep test data acquisition and data based design compared to plants operation of 20 to 30 years ago. Experience in Japan suggests that 81% of power-generation boilers are due to mechanical destruction of boiler materials, and that creep fracture is the major reason. In order to design safe power-generation plants, long term creep strength data are mandatory. In the case where no accurate creep data are provided, a higher safety margin is necessary and plants must be operated at lower vapour temperature to prevent creep degradation of boiler materials. Lower vapour temperature causes lower generation efficiency.

7 REFERENCE MATERIALS

Reference materials play an important role in enabling traceability, in allowing calibration of test equipment and ensuring confidence in measurement and testing data. Reference materials are defined in ISO Guide 35:2006 as:

> **Reference material (RM) -** Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process.

Notes:

1) RM is a generic term.

2) Properties can be quantitative or qualitative, e.g. identity of substances or species.

3) Uses may include the calibration of a measurement system, assessment of a measurement procedure, assigning values to other materials, and quality control.

4) A RM can only be used for a single purpose in a given measurement.

> **Certified reference material (CRM) -** Reference material characterised by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

Notes:

1) The concept of values includes qualitative attributes such as identity or sequence. Uncertainties for such attributes may be expressed as probabilities.

2) Metrologically valid procedures for the production and certification of reference materials are given in, among others, ISO Guides 34 and 35.

3) ISO Guide 31 gives guidance on the contents of certificates.

7.1 ISO GUIDELINES ON REFERENCE MATERIALS

The primary body in ISO responsible for reference materials is ISO/REMCO (the ISO Committee on Reference Materials). As with other ISO committees, membership is open to interested ISO member bodies as participating (P) or observer (O) members and to international liaisons. Its objectives are to carry out and encourage a broad international effort for the harmonisation and promotion of CRMs, their production and their application, and to be the global centre of excellence in relation to issues relating to reference materials. ISO/REMCO clients fall into four categories:

- users of CRMs, namely analytical/measurement and calibration laboratories;
- producers of reference materials;
- accreditation bodies, in particular with respect to accreditation of reference material producers; and

• other ISO Committees making use of the horizontal advisory function of REMCO.

ISO/REMCO is responsible for the ISO 30-35 series of guides related to reference materials detailed in Table 7.1.

Table 7.1 ISO Reference Material Guides

ISO Reference Material Guides
ISO 30 Terms and definitions used in connection with reference materials
ISO 31 Reference materials - Contents of certificates and labels
ISO 32 Calibration in analytical chemistry and use of certified reference materials
ISO 33 Uses of certified reference materials
ISO 34 General requirements for the competence of reference material producers
ISO 35 Reference materials - General and statistical principles for certification

7.2 REFERENCE MATERIALS AND CIPM CONSULTATIVE COMMITTEES

Reference materials form an essential part of the dissemination of metrological traceability between member institutes of the consultative committees and towards their customers. For example:

- **Hardness reference blocks** are well established as an essential part of the traceability chain for hardness measurements. As noted elsewhere, hardness is a procedural controlled material property, so that reference blocks are available for each test procedure (e.g. Rockwell, Brinell).
- **Dielectrics reference materials:** in spite of the wish to use Tissue Mimicking Materials (TMM), for example, in the measurements of the impact of mobile phone transmissions, reference materials are in fact liquids as being more reliable, available and consistent.
- **Acoustic reference materials:** in a manner similar to above, filled gels are used as TMM.
- **Thermal reference materials:** within CCT WG 9 projects are underway to establish comparative data. .
- **Magnetic reference materials:** these are available for the low magnetic permeability range based on dispersions of iron fillings in acrylic to provide a range of set values. Metal reference materials available with stated values are stainless steel (AISI Type 316), Naval brass (CZ114) and Ni/Al/bronze (DGS 1043).

7.3 REFERENCE MATERIALS AND THE INTERNATIONAL LABORATORY ACCREDITATION COOPERATION (ILAC)

ILAC is aiming to coordinate and harmonise internationally the accreditation of laboratories. One of the basic requirements is the proper use of reference materials, demanded for instance by ISO/IEC 17025 (General requirements for the competence of testing and calibration laboratories).

In addition to ILAC-G12 referenced in Section 6.2, several other ILAC guidelines exist that relate to the production and use of reference materials. One of the final objectives against which the aptitude for use of a Certified Reference Material (CRM) is evaluated is its contribution to the uncertainty of the analyses, made by means of the calibration performed with the CRM. The metrological quality of the analysis is its uncertainty. Other parameters may also be important (cost, speed, practicality, commutability), but these must be considered as subordinate to the assurance that the criterion of accuracy meets the level required for use.

Table 7.2 ILAC Guides related to Reference Materials

No.	Title
ILAC-G2:	Traceability of Measurement
ILAC-G4	Guidelines on Scopes of Accreditation
ILAC-G8:	Guidelines on Assessment and Reporting of Compliance with Specification
ILAC-G9:	Guidelines for the Selection and Use of Certified Reference Materials
ILAC-G12:	Guidelines for the Requirements for the Competence of Reference Material Producers
ILAC-G17:	Introducing the Concept of Uncertainty of Measurement in Testing in Association with the Application of the Standard ISO/IEC 17025
ILAC-G18:	The Scope of Accreditation and Consideration of Methods and Criteria for the Assessment of the Scope in Testing

The G9 guideline exclusively addresses the case in which CRMs are used for calibration. It discusses criteria with respect to chemical analyses by general methods, but the concepts can be applied to other areas of material testing. Demonstrating the quality of chemical analyses often implies demonstrating the quality of the CRM used for calibration. This point highlights the importance of accreditation of CRM producers.

7.4 REFERENCE MATERIALS AND THE JOINT COMMITTEE FOR TRACEABILITY IN LABORATORY MEDICINE (JCTLM)

As noted previously, the Joint Committee for Traceability in Laboratory Medicine (JCTLM) was established by CIPM, IFCC and ILAC. The JCTLM created two working groups:

- WG1 Reference Materials and Reference Procedures
- WG2 Reference Laboratory Networks

JCTLM WG1 was charged with establishing a process for identifying, reviewing against agreed upon criteria, and publishing List(s) of Higher Order Certified Reference Materials and Reference Measurement Procedures required for industry compliance with the EC In Vitro Directive regarding *in vitro* diagnostic medical devices. Nominated reference materials and measurement procedures are categorised according to the criteria described in ISO 15193 and ISO 15194. Two Lists of Higher Order Reference Materials and Reference Measurement Procedures are published so far:

- List I. Certified Reference Materials and Reference Measurement Procedures for well defined chemical entities or internationally recognised reference method-defined measurands. Reference Materials and Measurement Procedures included in this category are those that provide values that are traceable to the SI units; e.g., Electrolytes, Drugs, Metabolites and Substrates, Non-Peptide Hormones, Enzymes and some Proteins.
- List II. Reference Materials (e.g. reference materials for Blood Typing, Coagulation Factors, Microbial Serology, Nucleic Acids, and some Proteins) that are value assigned using an internationally agreed upon protocol. The values of the measurands in the reference materials on this List are not SI-traceable and/or no internationally recognised reference measurement procedures exist. List II also contains a group of purified

substances that due to the absence of reference measurement procedures should not be directly used for calibration unless commutability is established.

In submitting reference materials for evaluation and adoption, traceability is allowed to:

a. SI units

b. Other standard (such as ISO).

c. World Health Organisation

Comment: The work programme of the Joint Committee for Traceability in Laboratory Medicine is heavily focussed on reference materials and reference methods. "The aim of the JCTLM is to support world-wide comparability, reliability and equivalence of measurement results used for assessing and monitoring human health status and for facilitating national and international trade of in vitro diagnostic devices". Some equivalence with the situation regarding materials metrology is apparent.

7.5 USE OF REFERENCE MATERIALS IN PROFICIENCY TESTING

Several commercial and governmental organisations organise proficiency testing. To be able to make definitive statements on the proficiency of a laboratory, the organiser of the proficiency test must rely on the homogeneity and stability of the test materials distributed to the participating laboratories. For this reason, the test materials need to be assessed prior to the proficiency test. De facto, this requires that the test materials qualify as 'reference materials'. The use of reference value is recommend for assessing the analytical competence of the participating laboratories, because the measurement results compared to the reference value allow the bias to recognised with respect to the "true value", and consequently the sources of the bias to be evaluated. A problem may lie in the fact that even when some NMIs support the proficiency test by providing reference values for those proficiency tests organised by specialised companies or other materials research institute, the reference value is not internationally accepted as a true basis of comparable data. In other words, comparability of accredited laboratories is not guaranteed even by proficiency testing.

7.6 AVAILABILITY OF REFERENCE MATERIALS WITH CERTIFIED PROPERTIES

Reference materials with certified material properties are available from several sources. Suppliers with a broad range of reference materials in this field are for instance NIST, KRISS, IRMM, and BAM. In addition, a large number of companies provide reference materials for one or a few particular material areas.

NIST:

- Mechanical properties (fracture toughness of ceramics, impact toughness of steels, Hardness and micro-hardness standards, Young's modulus, yield strength and elongation, abrasive wear, adhesion properties)
- Morphological properties (particle size, electrophoretic mobility, surface area and porosity, phase composition, layered and coated materials, lattice spacing)
- Thermal and thermodynamic properties (critical temperatures, thermal expansion, thermal conductivity)
- Optical properties (reflectance, refractive index)
- Electrical properties (resistivity, conductivity, critical current)

- Corrosion (glass chemical resistance)

KRISS

- Mechanical properties (hardness)
- Morphological properties (thin films, crystallographic orientation, surface area)
- Thermal and thermodynamic properties (thermal conductivity, critical temperatures)
- Optical properties (colour reference materials, refractive index)
- Electrical properties (resistivity)

IRMM:

- Mechanical properties (Charpy impact toughness, scratch resistance, creep resistance, tensile strength, powder shear stress)
- Optical properties (colour reference materials)
- Thermal properties (thermal conductivity, thermal diffusivity, cold filter plugging point)
- Morphological properties (particle size, surface area, film thickness, lattice spacing)

BAM:

- Morphological properties (particle morphology and porosity, multi- and single layers)
- Optical properties (colour reference materials)
- Mechanical properties (tribological reference test samples)

Some typical property/material combinations are listed in Table 7.3

Table 7.3 Typical Reference Material/Property Data

Property / Material	Mean Value	Uncertainty
Thermal conductivity / resin bonded glass fibre board [(W/(m.K)]	$\lambda = 0.0293949 + 0.0001060 \times T + 2.047 \times 10^{-7} \times T^2$	0.0028 between –10 °C and 50 °C at 95% confidence
Thermal diffusivity / glass-ceramic [$m^2/s.10^6$]	$\alpha = 4.406 - 1.351.10^{-2}\, T + 2.133.10^{-5}\, T^2 - 1.541.10^{-8}\, T^3 + 4.147.10^{-12}\, T^4$	6.5 % between 298 K and 1025 K at 95% confidence
Creep / metal at 600 °C at 160 MPa	Creep rate at 400 h = $72\ .10^{-6}\ h^{-1}$	$5.10^{-6}\ h^{-1}$
	Time to creep strain of 2 % = 278 h	16 h
	Time to creep strain of 4 % = 557 h	30 h
Tensile strength / Nimonic 75	0.2 % proof stress = 300 MPa	7 MPa
	0.5 % proof stress = 318 MPa	16 MPa
	Ultimate tensile strength = 750 MPa	13 MPa
	Elongation to fracture = 40.9 %	0.9 %
	Reduction in area = 60 %	4 %
	Young's Modulus = 206 GPa	21 GPa
Charpy V-notch impact / steel at 20 °C	Absorbed energy = 58.7 J	1.5 J at 95% confidence

Comment: Although, not the majority, a significant number of certified reference materials exist that cover mechanical, thermal, electrical, morphological and optical properties.

8. PROCEDURES FOR DETAILED PROPERTY REVIEWS

At the first meeting of the ad-hoc group, Task Groups were established related to five material property areas defined as follows.

Table 8.1. Agreed list of properties assigned to Task Groups.

PROPERTY AREA	DETAILED ASPECTS	TASK LEADER	TASK MEMBERS
Mechanical	Hardness	**Graham Sims / Bryan Roebuck**	Junichi Kasai, Richard Kayser, Alexandro Germak, Gun Woong Bahng
	Modulus		
	Strength		
	Toughness		
	Fatigue		
	Creep		
	Viscosity		
Thermophysical (Phys-chem)	Conductivity	**Tetsuya Baba**	Wolfgang Buck, Philippe Charlet
	Diffusivity		
	Expansion		
	Specific Heat		
	Emissivity		
Composition and micro-structural	Grain size / boundaries	**Richard Kayser**	Bryan Roebuck, Juergen Lexow, Yoshito Mitani
	Phase		
	Porosity		
	Texture		
	Particle size		
	Defects		
Functional	Electrical	**Graham Sims**	Carlos Achete, (Markys Cain, Bob Clarke)
	Optical		
	Magnetic		
	Thermo-electric		
Electrochemical		**Juergen Lexow**	Bryan Roebuck, (Alan Turnbull)

In addition, a list was prepared of the aspects that were required to be assessed for each property, as given below in table 8.2. This list was assembled from a brainstorming session and descriptors added later. It was important for the success of the reviews that all Task Groups had the same interpretation and understanding of the questions and terms used. The information collected was reviewed at the second meeting of the ad-hoc group. This information was subsequently replaced by a second review aimed specifically at assessing the impact of the need for traceability and comparability in each case. The proforma developed for the second stage is given in table 8.3. The results of this stage of the analysis were reported and discussed at the NIST meeting in December 2006.

Table 8.2 Agreed list of questions on each property.

ASPECT	PROPERTY (e.g. Modulus)
Scope	*Short description of property and measurement methods/techniques*
Material Category	*Metal, ceramic, polymer, composite, rubber, etc.?*
Material State and Scale	*Liquid, micro, particle, bulk, film, nano, surface?*
Regulation Need	*Is this property a directly or implied requirement in any regulative document? (e.g. state law, EU directive, industry (CAA, DNV, Lloyds) regulation etc.)*
User needs	*What are the user requirements?* *(excluding above regulatory aspects)*
Accreditation needs	*Is this property needed in any accreditation procedure?*
Comparability	*Is there a need for comparability of data?* *(e.g. if required in design codes, when more than one code and/or test method exist for the same application, such as pressure vessels)*
Need for intercomparison	*Should the WGMM initiate interlaboratory comparisons or recommend future exercises?*
Economic impact studies	*Have any economic studies been undertaken of the impact of incorrect or high scatter measurements of this property?*
Number of existing methods	*How many "standardised" or accepted methods are used to measure this property?*
Standardisation situation	*What standards exist and do they include precision data? (please attached any data)*
NMI/MRI activities	*Is there any activity in this property measurement at these organisations?*
R & D phase?	*Is there any basic research into this property and/or new measurement techniques*
Traceability issues	*What is the traceability route for this property?*
SI units relevant	*Which SI unit (if any) is relevant?*
CCs coverage	*Is this property covered by any existing CC?*
Prior studies (method specific)	*Have any prior studies been undertaken for this property? (please attached any data)*
Method comparability	*Have any prior comparability studies been undertaken for this property? (please attached any data)*
Standard test machines	*Do standard test machines exist for these measurements?* *Have machines been compared?*
Calibration – reference materials	*Do calibrants/reference materials exist for this property?*
Uncertainty	*Have any uncertainty budgets been developed for these measurements?*

Table 8.3 Proforma (b) for property evaluation

Task Group No. < >	Issue / Evidence	Comment / Plan
Material Property	e.g. optical, thermal conductivity, magnetic, dielectric, acoustic	
Is the material property important? (e.g. Does it have importance in trade, regulations, etc.?)		
Is there a problem with consistency / comparability of the measured material property?		
Is the problem caused by an unaddressed need for traceability?		
What other aspects are relevant to the problem?		
Are there important consequences if this problem is not addressed? (e.g. Can the financial or regulatory impact of the issue be estimated?)		
What is the way forward? (e.g. Is the property covered by an existing CC? (Inform and offer to support any work they initiate?), Is it suitable for VAMAS, a standards development organisation, or a regional metrology organisation (Euromet) activities?, Is work in conjunction with CIPM and/or ILAC necessary?, Is a reference material needed?).		

This information was subsequently replaced by a second review aimed specifically at assessing the impact of the need for traceability and comparability in each case. The proforma developed for the second stage is given in table 8.3. The results of this second stage of the analysis were reported and discussed at the LNE meeting in May 2007.

9 TASK GROUP 1 – MECHANICAL PROPERTIES

This area covers mechanical properties, such as hardness, toughness, modulus, strength, creep and fatigue.

9.1 MODULUS

The modulus of elasticity is an intrinsic property and therefore its value should not be dependent on the detailed procedures (e.g. all the measured properties are directly related to length (i.e. specimen dimensions, gauge length, deformation) or mass (i.e. applied force)). However, the evidence assembled suggests that this measurement, even for a metal at room temperature, cannot be conducted at a satisfactory level that is "fit-for-purpose". The evidence includes the original VAMAS data (see figure 14.2), the Proficiency testing data and the experience of NPL regarding the Formula 1 measurements. The SI units involved of length and mass cover two CCs areas of responsibilities.

> **Comment: There is a clear need to demonstrate for this intrinsic property that "acceptable" uncertainties can be obtained. It is proposed that this area should be used for a pilot study.**

9.2 HARDNESS

The measurement of hardness is probably the best example of a procedural controlled material property, and one that might have been least likely to be recommended from this review as relevant to CIPM. It is one of the most frequently referenced **controlled** properties. Several alternative test methods have been standardised, such as, as Rockwell, Brinell and Vickers, which are well established and are not convertible from one scale to another. For historical reasons, thought to be associated with the hardness of knife-edges in mass balances, these measurements have been associated with CCM, with the usual key comparisons being undertaken. On the CIPM web-site 8 key and 8 supplementary comparisons are listed. A comprehensive series of reference materials or hardness blocks exists to enable calibration.

> **Comment: Hardness is for historical reasons already within CCM, but developments into micro and nano-hardness measurements will increase the existing trend that these measurements are made in research laboratories and industry by materials specialists not mass/force specialists. Progress and needs in these newer measurements should be reviewed by a CCM/VAMAS group, including current EU and VAMAS projects**.

9.3 TOUGHNESS

Toughness has been shown in the past to be a critical property in structural applications, from the Comet aircraft fractures to brittle cracking of railway lines. It is of particular concern as fracture is often sudden and unexpected, and the life and economic costs of failure are high. Fracture toughness is a complex property that relates to several SI units (e.g. mass, length, time). Several round-robins have been undertaken in this area.

> **Comment: This property is of obvious importance as shown by its technical aspects, but also the fact that the property is currently under extensive study (i.e. research and round-robin projects) and has reference materials available.**

9.4 ULTIMATE STRENGTH

The ultimate strengths, or a safety-factored value, are the principal properties used in design to assess strength-dominated designs. There are few reference materials available, and unlike moduli values they are dependent on the procedures used.

9.5 CREEP

This is an important property, especially for polymeric materials at room temperatures and for metals at high temperatures in ensuring reliability and safety. It is important to maintain the applied load, traceable to the kilogram, over a long period of time.

Recently, there is a trend to use power plants longer than the designed life to reduce environmental contamination and to save resources. For the decision making how long the power plant can be used further, e.g., estimation of residual life of high temperature materials components in boiler, creep data plays a very important role.

9.6 FATIGUE

Similar arguments can be made as for creep, but of increased cost due to the need to cycle the load, or strain, in a specified manner. No current round-robins known.

9.7 IMPACT

Impact is a well-established mode of testing, especially for QA purposes. Dependent on the procedure being used which includes bending (Charpy, Izod) and tension. Higher rate tests includes gas guns and shock tubes. Increasingly, there is interest in measuring conventional properties (e.g. tension, compression and shear) at high strain rates to use in crash modelling predictions. Work undertaken in EU research programme on uncertainties.

9.8 RHEOLOGY

The rheological properties of melts, both metallic and plastic, are important for the efficient and consistent processing of materials through casting and injection process routes. Increasingly, modelling capabilities are being developed that require accurate input materials rheological data.

9.9 CONCLUSIONS FROM TG1 - MECHANICAL PROPERTIES

- **Examples of top level regulations were reviewed (e.g. EU Pressure Vessels Directive) which require information on underpinning materials properties, such as strength, corrosion resistance and microstructure.**
- **Measurements are often dealt with by more than one CC (e.g. CCM and CCL for modulus), so that coordination of activities would be beneficial.**
- **The main impact of incorrect measurements (e.g. modulus, including large uncertainty effecting A and B design values and incompatible data from different techniques) is in inefficient design, inapplicability to apply new approaches and reduced reliability/increased chance of failure.**
- **Modulus is an important design property that as an intrinsic property should be the subject of a pilot study to establish improved uncertainty in its measurement.**

10 TASK GROUP 2 – THERMOPHYSICAL PROPERTIES

This area covered thermal conductivity, diffusivity, expansion, specific heat, emissivity and glass transition temperature.

10.1 THERMAL CONDUCTIVITY

Traceability is well established for low conductivities, where reference materials exist. For medium to high conductivities, a demand for reference materials was identified, but few are available on the market. A need is especially expressed for the characterisation of ceramics and new polymers. Manufacturers of instruments are also requiring reference materials for the calibration of their instruments. For established materials, no specific problem of metrological traceability was identified. Reference methods exist (available at NMIs), but a lack of reference materials was observed.

> **Comment: For new materials linked to micro and nano scale, a traceability problem has been identified. Emergent sectors (electronic and bio) have expressed their needs for conductivity measurements at these scales.**

10.2. THERMAL DIFFUSIVITY

Basic information for thermal design (e.g. Production control in metallurgy and ceramic industries) depends on reliable diffusivity data and testing laboratories accredited for thermal diffusivity measurement need traceability.

> **Comment: Thermophysical properties are covered by CCT WG9 - "Thermophysical properties".**

10.3. THERMAL EXPANSION

Supply of certified reference materials is usually the traceability route for thermal expansion (suppliers - NMIJ (Japan), NIST (USA), PTB (Germany)). Uncertainty budgets have been developed for these measurements by NMIJ, Japan

> **Comment: Thermophysical properties are covered by CCT WG9 - "Thermophysical properties".**

10.4. SPECIFIC HEAT CAPACITY

Cp of heat exchanger fluids is necessary for determining the efficiency of thermal engines and of building materials for fire protection. Uncertainty of 5 % is often sufficient. Accredited testing laboratories for Cp testing need traceability.

> **Comment: Thermophysical properties are covered by CCT WG9 - "Thermophysical properties".**

10.5 GLASS TRANSISTION TEMPERATURE

Measurement of the glass-transition temperature is an important method for checking the cure state of reinforced plastics, especially high performance polymer matrix composites and is measured by changes in many other properties such as dielectric, thermal expansion, stiffness, acoustics (ultrasonics), as a function of temperature. Comparability, as well as traceability to SI, of the different methods is of concern to industry and needs attention.

Comment: There is a need to show that these alternative methods all measure the same glass transition temperature and a pilot study should be proposed. Improved traceability and calibration of temperature, in particular, length and force should be included.

10.6 CONCLUSIONS FROM TG2 – THERMOPHYSICAL PROPERTIES

- **There are extensive needs for metrology of thermophysical properties from science, industry, energy conservation, safety, and trade.**
- **Thermophysical properties are inherent and represented by SI-traceable derived units.**
- **Temperature dependence of thermophysical property is essential information.**
- **Thermophysical properties are covered by CCT WG9 "Thermophysical properties".**
- **Thermophysical properties can be a good area of collaboration between an existing CC and material metrology activity in areas such as reference materials, thermophysical properties for design and for advanced materials, such as nanomaterials.**

11 TASK GROUP 3 - COMPOSITION AND MICRO-STRUCTURAL

This area covers grain size / boundaries, phase, porosity, texture, particle size and defects.

11.1 GRAIN SIZE

Quantitative microscopy techniques are the primary methods for measuring grain size in conventional materials. Methods for very fine grain materials (submicron) include x-ray (systematic peak broadening) and transmission electron microscopy techniques.

Comment: There is no evidence to suggest that customers have significant metrological issues related to measuring grain size. No obvious role for the CIPM.

11.2 PHASE

Routine methods include x-ray diffraction for crystalline materials augmented by electron microscopy methods. Non-routine methods include neutron diffraction and synchrotron-based methods. There is no evidence of a problem with consistency / comparability. Users need low cost, ease of use, user-friendly data interpretation software, and databases of known quality to identify unknown materials.

Comment: There is no evidence to suggest that customers have significant metrological issues related to measuring phase. No obvious role for the CIPM.

11.3 POROSITY

Numerous methods exist for measuring porosity in different materials under different circumstances, e.g., gas adsorption, mercury intrusion, fluid flow, neutron scattering, x-ray scattering, electron microscopy, NMR, others.

Comment: Important for standards development organisations, VAMAS and developers of reference materials. No obvious role for the CIPM at this time.

11.4 CRYSTALLOGRAPHIC TEXTURE AND ORIENTATION

Crystallographic texture measurements include uniaxial - typically developed in thin films and rods; and three dimensional, typically developed in rolled sheets of metal cases. There is little known activity in the development of either documentary standards or reference materials for texture per se and no evidence of a problem with consistency / comparability.

This property is important in ensuring, for example, the correct drawing characteristics for steels, also in measuring inter-granular corrosion for gas-cylinder approval. Different techniques can be used to determine grain orientation, with non-compatible result presentation. No reference images or materials are available for calibrating measurements

Comment: May be important for standards development organisations, VAMAS and developers of reference materials. No obvious role for the CIPM at this time

11.5 PARTICLE SIZE

Dozens of methods exist for measuring particle size under various conditions and in various states. Reported particle size often depends on the technique used. No problem with traceability at this time, but if a need for lower uncertainties develops, especially for nano-scale particles, traceability could become a more important issue.

Comment: Important for standards development organisations, VAMAS and developers of reference materials: property covered under the CCL. However, a case was made for NMI experts to meet, as particle size measurement has not been covered by any CC to date.

11.6 DEFECTS

There are numerous measurement needs related to defects, expressed by multiple industries. Defects are called out as a key challenge for nanoelectronics, photonics, and magnetics. User needs include assessment of defect type, number density, and statistical characteristics of defect populations, into two common themes: sensors and in-line monitoring. There is no evidence of regulatory or accreditation needs or drivers at this time. There is little known activity in the development of either documentary standards or reference materials for defects per se, especially for crystalline defects. Numerous comparisons of different techniques exist, but usually semi-quantitative. No reference images or materials are available for calibrating measurements.

Comment: May be important for standards development organisations, VAMAS and developers of reference materials. No obvious for the CIPM at this time

11.7 CONCLUSIONS FROM TG3 - COMPOSITION AND MICRO-STRUCTURAL

- **The conclusions generally suggested that while there was a role for VAMAS in improving the measurements, there was "no compelling reason" for CIPM involvement**.

12 TASK GROUP 4 - FUNCTIONAL PROPERTIES

This area covered dielectric (CCEM), optical (florescence), magnetic (CCEM), acoustic (CCAUV) properties; and some additional comments on thermal (CCT).

12.1 ELECTRICAL PROPERTIES

Traceable measurement of dielectric properties is stipulated in international standards. Health and Safety standards relating to RF interactions with humans (e.g. RF generated by mobile phones) and in 2008 the EC Physical Agents (EMF) Directive will require all employers to abide by these standards. There is a more general problem of supplying good quality dielectric data as input to EM field-modelling programmes: these days the algorithms are of good quality and the predicted performance of component/system designs is often limited rather by the large uncertainties and/or errors in the dielectric data measurement. Comparisons between labs often exhibit discrepancies, which exceed joint estimated uncertainties - sometimes to a considerable extent.

> **Comment: A microwave measurement comparison is currently being run by the Euromet High Frequency Electromagnetics sub-field, which may highlight some of these problems (internationally it comes under the remit of CCEM). Growing demand from industry for better measurements may trigger a need for a more comprehensive programme of international metrology here.**

12.2 MAGNETIC PROPERTIES

Measurement of specific total loss requires a magnetic circuit and the realisation of this circuit is a known problem. New materials are measured using a different circuit and this makes comparisons difficult. The electrical traceability for standard waveform conditions at low frequencies is available. For operational waveforms and higher frequencies problems occur.

> **Comment: Without traceable measurements to develop the materials and optimise device performance considerable energy will be wasted. With the move to more electric transport being driven by environmental issues the impact is large and far-reaching.**

12.3 OPTICAL PROPERTIES

Needs are developing for reference materials for gloss, apparent colour and texture as it appears optically and also for properties such as transmission and colour. Although there are established methods, there do not appear to be round-robin evaluations of the precision.

12.4 ACOUSTIC PROPERTIES

Relevant material acoustic properties include speed of sound and the attenuation/absorption/scattering behaviour. It is noted that tissue-mimicking (TMM) or tissue-like materials use water-based gelatine, loaded with scattering materials to generate the 'correct' properties rather than solid materials. Comparisons undertaken to date have been in the regional areas, rather then offered for worldwide participation.

12.5 CONCLUSIONS FROM TG4 - FUNCTIONAL PROPERTIES

- **There are materials issues where cooperation with existing CC work would be beneficial. For example, the large uncertainty in material dielectric data is limiting predicted performance of systems and components, rather than the algorithms used – current work in CCEM may highlight some of these issues so that a joint approach could be of benefit. Equally, magnetic material property measurements are made under standard conditions that are not representative of actual use.**

- **The measurement of the glass-transition temperature is an important method for checking the cure state of reinforced plastics, especially high performance polymer matrix composites and is measured by changes in many other properties such as, dielectric, thermal expansion, stiffness, acoustics (ultrasonics), as a function of temperature. Comparability, as well as traceability to SI, of the different methods is of concern to industry and needs attention.**

13 TASK GROUP 5 - ELECTROCHEMICAL PROPERTIES

13.1 CORROSION PROPERTIES

This will strongly influence the corrosion of the material as whole. Resolving this question will lead to more reliable corrosion measurement and improved corrosion protection. Related CCs are CC-Length, CC-Electricity and Magnetism, CC-Mass and Related Quantities and CC-Amount of Substance - Metrology in Chemistry.

Comment: The existing knowledge and standards do not sufficiently allow us to understand the grain orientation dependant corrosion rates (e.g. surface energy, etching rate and surface chemistry are often grain orientation dependant).

13.2 CONCLUSIONS FROM TG5 - ELECTROCHEMICAL PROPERTIES

- **The importance of local microstructure on corrosion performance was highlighted, which links with prior comments on orientation measurements under TG3. The economic impact of poor corrosion measurements is high and new material development is discouraged. Research work was suggested for VAMAS (related to microstructure), while trade and regulatory issues were proposed to be more appropriate to ILAC. Additional needs are developing in measurements on fuel cells and photovoltaics, where there is a need to measure the properties locally at the nanoscale.**

14 THE ISSUES: MEASUREMENTS, TRACEABILITY AND STANDARDS

14.1 MEASUREMENTS AND INTERCOMPARISONS

In most areas of metrology, the concept of traceability to established national or international standards is well understood. The SI (Système International) provides a coherent set of well-defined units, which provide a common language for expressing and understanding the results of measurements. The NMIs maintain standards according to their national needs and the equivalence of these standards has been established through the mechanisms of the CIPM Mutual Recognition Arrangement: key intercomparisons, mutual review of claimed capabilities and regional assessment of NMI quality systems.

The result of this methodology is that NMIs can generally demonstrate excellent agreement in intercomparison exercises in these classical fields and they pass this metrological confidence on to accredited laboratories which are in turn required to demonstrate the traceability of their results. Figure 14.1 shows the results of measurements of the length of a 175-millimetre gauge block performed by eleven NMIs between September 1999 and June 2001. All the results agree within 1 part in 10^6 and with one exception the agreement is better than 3 parts in 10^7. This impressive result reflects a very thorough and careful approach to the relatively straightforward measurement of a length standard.

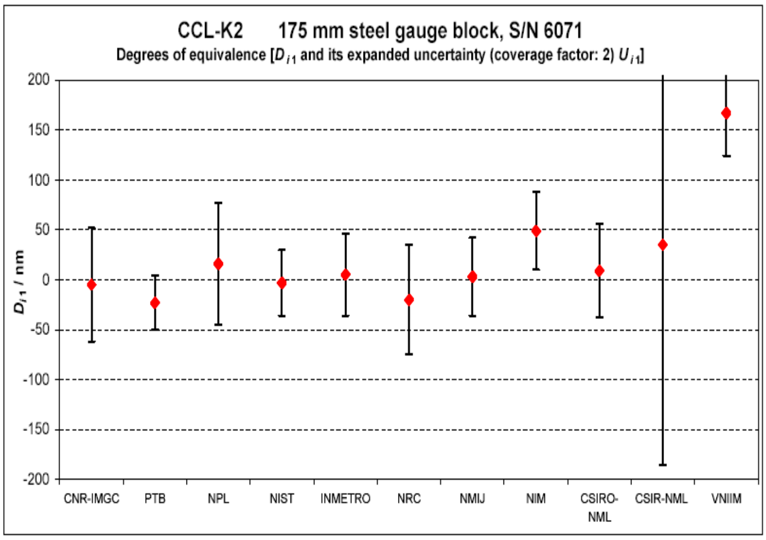


Figure 14.1. Results of international comparison of 175-mm gauge block [11]

Similar exercises to evaluate the ability of laboratories to measure materials properties are often less impressive. Figure 14.2 illustrates the results of a national intercomparison exercise in the UK to determine Young's Modulus. The spread of the results from 25 laboratories for a simple modulus measurement show a total spread of about 40%. There are many possible reasons for this enormous discrepancy between laboratories, but the contrast with the level of agreement obtained by NMIs comparing results for the calibration of a length standard could hardly be more marked.

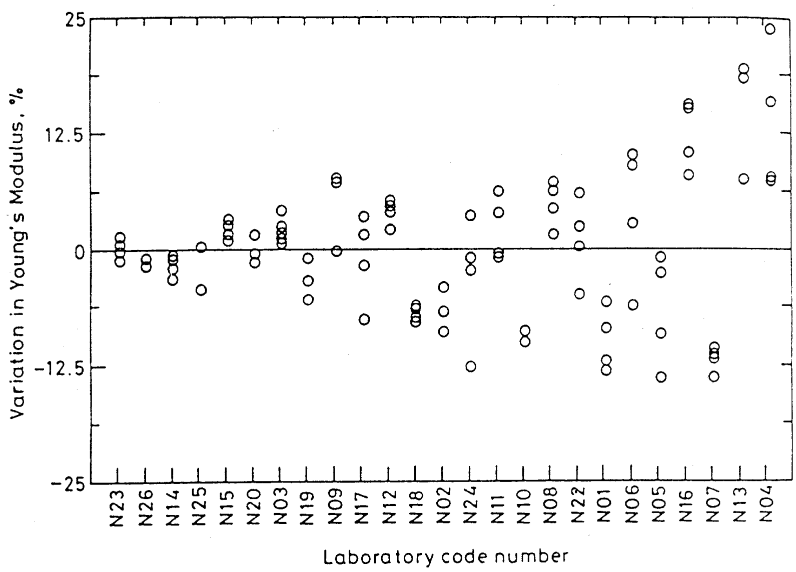


Figure 14.2. Results of round robin determination of Young's Modulus [12]

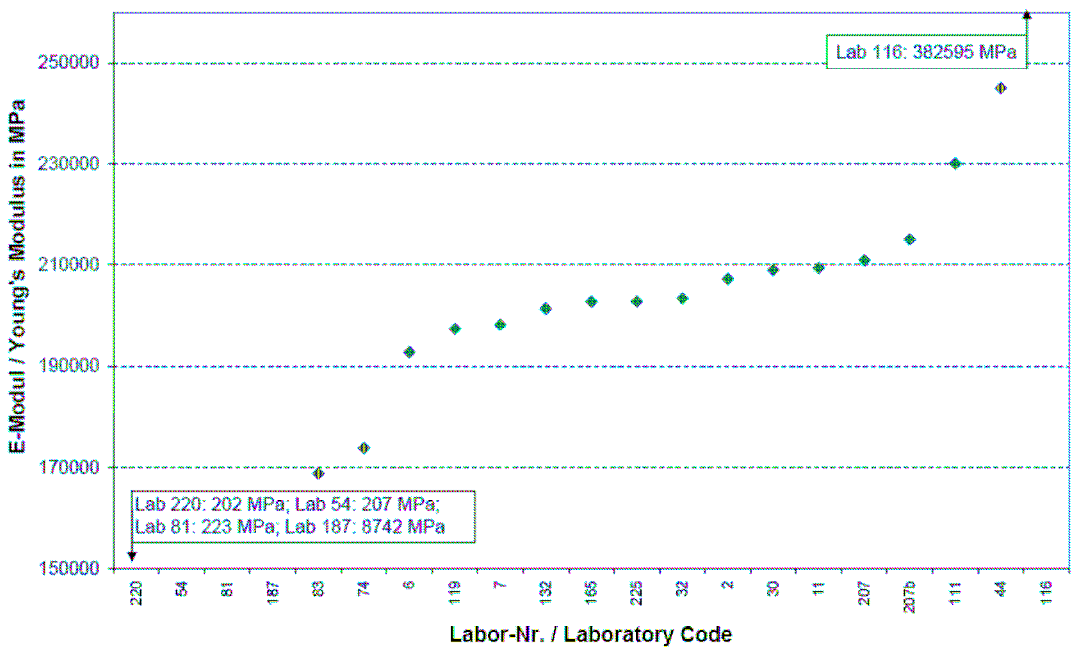


Figure B4: Stahl-Luftfahrtqualität: E-Modul; Mittelwerte aller Laboratorien
Steel Aircraft-Quality: Young's modulus; mean values of all laboratories

Figure 14.3 Results for Young's Modulus measurements at different sites [13]

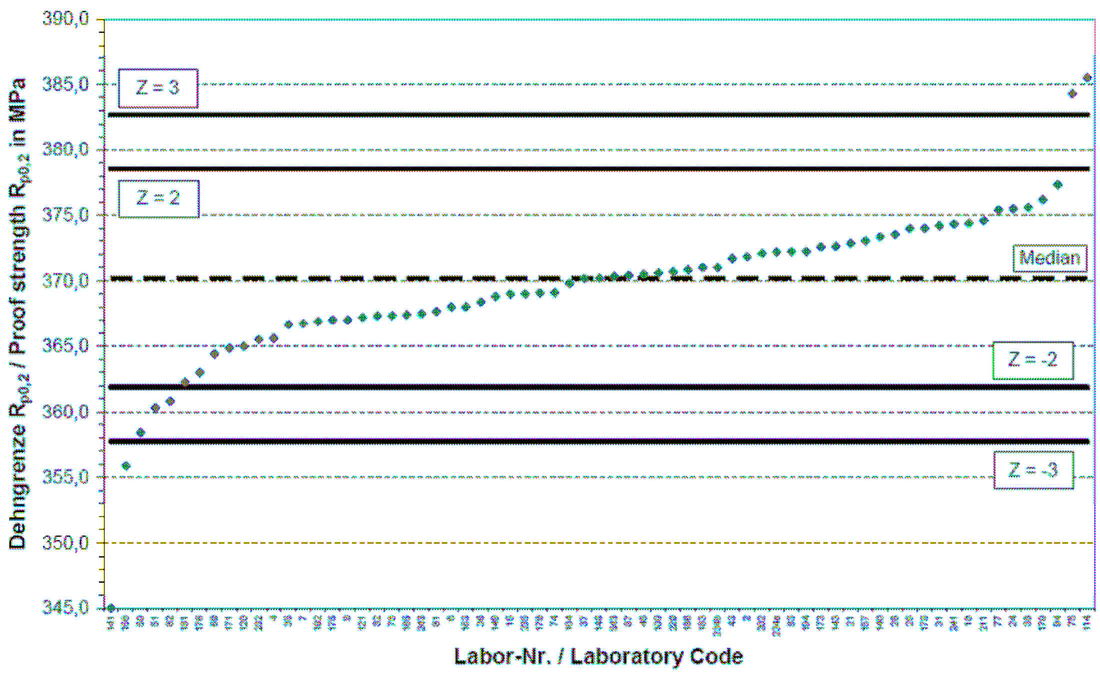


Figure A1: 0,2%-Dehngrenze; Mittelwerte aller Laboratorien / 0,2% Proof strength; mean values of all laboratories

Figure 14.4 Results for Proof Strength measurements at different sites [13]

This degree of uncertainty is also shown by data available from proficiency tests. For example, data from the IfEP website regarding a modulus trial showed results varying from 170 to 230 GPa, even after excluding obvious errors probably due to unit conversion (e.g. 210 MPa rather than GPa reported, also some very high values reported). The key point here is that this figure is not an isolated example and similar discrepancies are regularly observed, even when great care is taken to eliminate obvious sources of error.

A comparison of dynamic mechanical analysis (DMA) and differential scanning calorimetry (DSC), which are both routine methods used to measure the glass transition temperature (T_g) of polymers, is shown in figure 14.5. It is noted that both techniques show good repeatability and DSC good reproducibility, but DMA had poor reproducibility. In addition, other data provided in confidence, indicated a spread of 25% in the means of stiffness and strength values and more than a factor of two in the means of thermal analysis data.

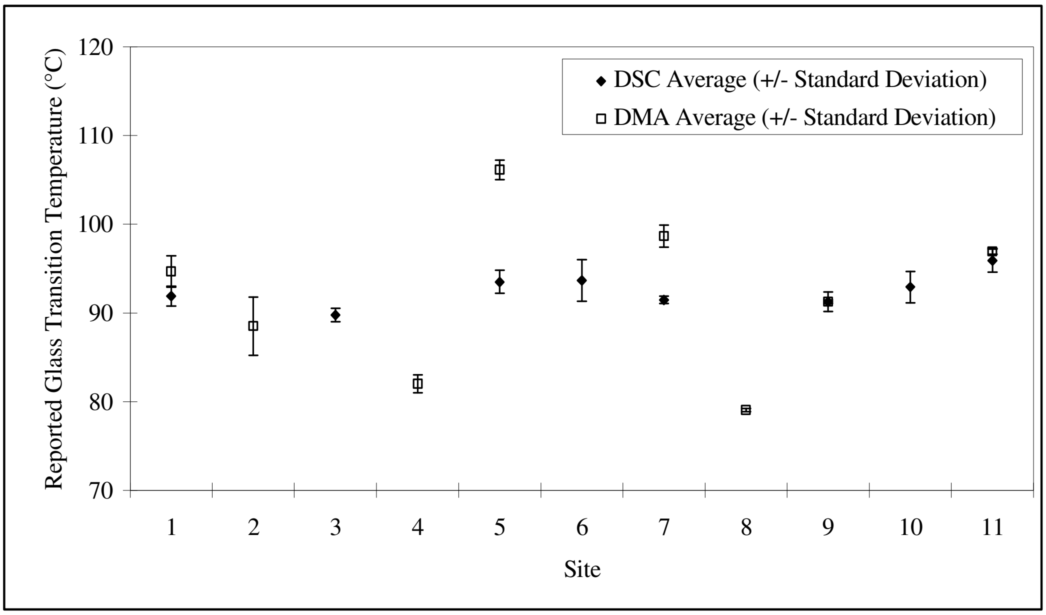


Figure 14.5 Results of round robin for glass transition temperature, T_g [14]

Other proficiency data for intrinsic properties show a range of 25% of the mean for individual data sets for mechanical tests, but over 100% for measuring T_g by monitoring thermal expansion.

14.2 INTRINSIC AND PROCEDURAL PROPERTIES

It is apparent that even for intrinsic properties, such as Tensile Modulus, that the scatter recorded is often unacceptable. In these cases (e.g. thermal expansion), the measurement procedure itself should not effect the measured result. The results maybe by many other parameters associated with the materials such as shape, surface condition, composition, grain, etc., an approach similar to that followed for procedural properties is recommended. Figure 14.2 shows potentially a good example since Young's Modulus is an intrinsic property. Usually, data reported in papers regarding mechanical properties, such as strength, fatigue, creep, show that the data has a scatter around several %, which is relatively large compared to other physical measurements.

For procedural properties, it is important to calibrate the procedure itself. This is necessary to guarantee the data obtained according to ISO or equivalent standards and that their comparability is supported by calibration of the procedure with CRMs. The uncertainty of test result should also be evaluated by CRMs. However, in many cases, CRMs are not available for these procedural material properties.

14.3 TRACEABILITY

The International Vocabulary of Metrology (VIM) [9] defines metrological traceability as the *"property of a measurement result whereby the result can be related to a stated reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty."* While this definition can be straightforwardly applied in a calibration laboratory, there is not normally a single chain of calibrations for the measurement of materials properties.

In a footnote, however, the VIM adds that *"for measurements with more than one input quantity in the measurement model, each of the input quantities should itself be metrologically traceable and the calibration hierarchy involved may form a branched structure or a network."* This is normally, if not always, the case for materials metrology, and it is essential to establish reliable traceability for the applied stimulus and the measured resulting effect (see figure 14.6) as well as for measurements of any other quantities that may influence the final result. This result may also be affected by the measurement procedure and by the state of the sample.

In the case of Young's modulus, for example, the stimulus takes the form of an applied load or stress and the measured effect is the resultant strain. The traceability of the stress will be established through a calibrated load cell and measured specimen cross-section; while determining the strain involves traceability of the measurement of the change in length of the measured gauge length. This may not be sufficient to ensure repeatable results, unless a common procedure is used on identically prepared specimens.

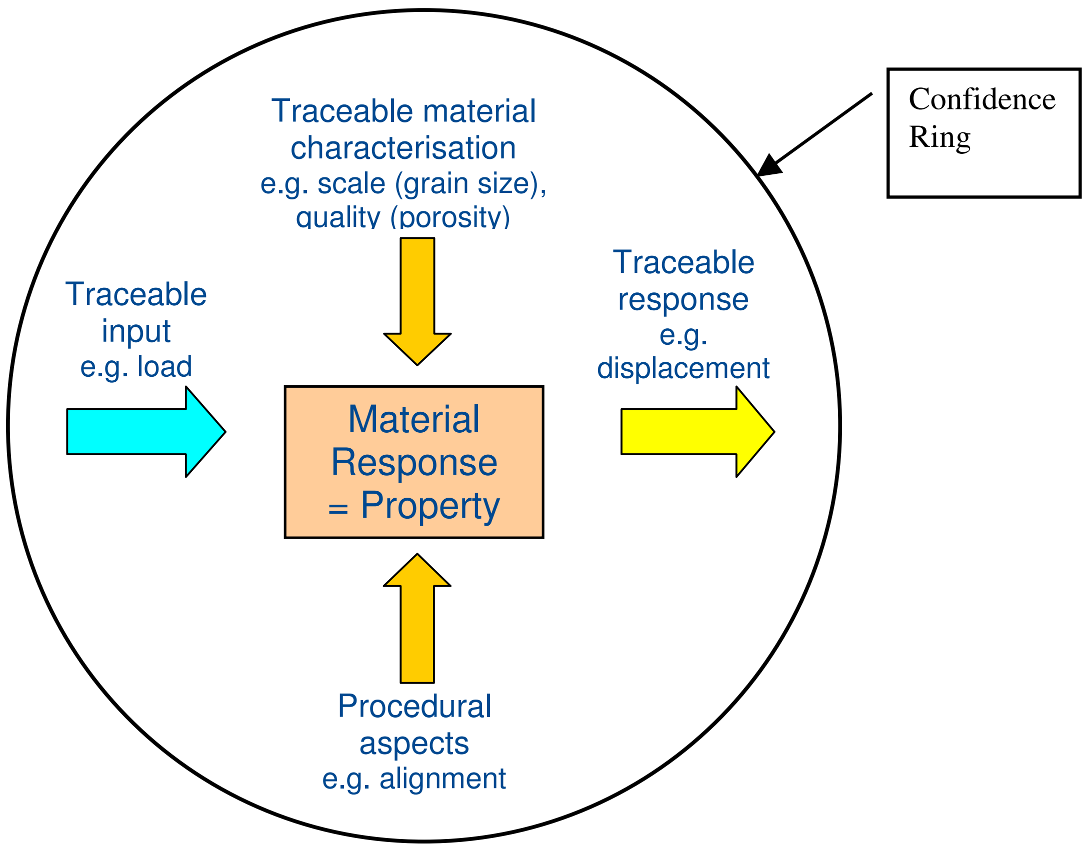


Figure 14.6 Confidence ring for material property measurement – note that separate traceability for applied stimulus, response and material characterisation.

14.4 UNITS

It is also an interesting point that modulus is normally expressed in gigapascals (GPa). Although dimensionally correct, the Pa is the unit of pressure and this practice may suggest traceability to a pressure standard. The SI brochure [15] suggest that *"a derived unit can often be expressed in different ways by combining base units with derived units having special names. … This, however, is an algebraic freedom to be governed by common sense physical considerations; in a given situation some forms may be more helpful than others. … In practice, with certain quantities, preference is given to the use of certain special unit names, or combinations of unit names, to facilitate the distinction between different quantities having the same dimension."* In the case of modulus measurements it is thus preferable to express results in terms of MN/m^2 (as it is the traceability of the applied force which can be established), although the use of GPa is also dimensionally correct. The modern preference for GPa may be that it avoids the use of a superscript, and is therefore easier and neater to use when typing on computers, especially in Tables.

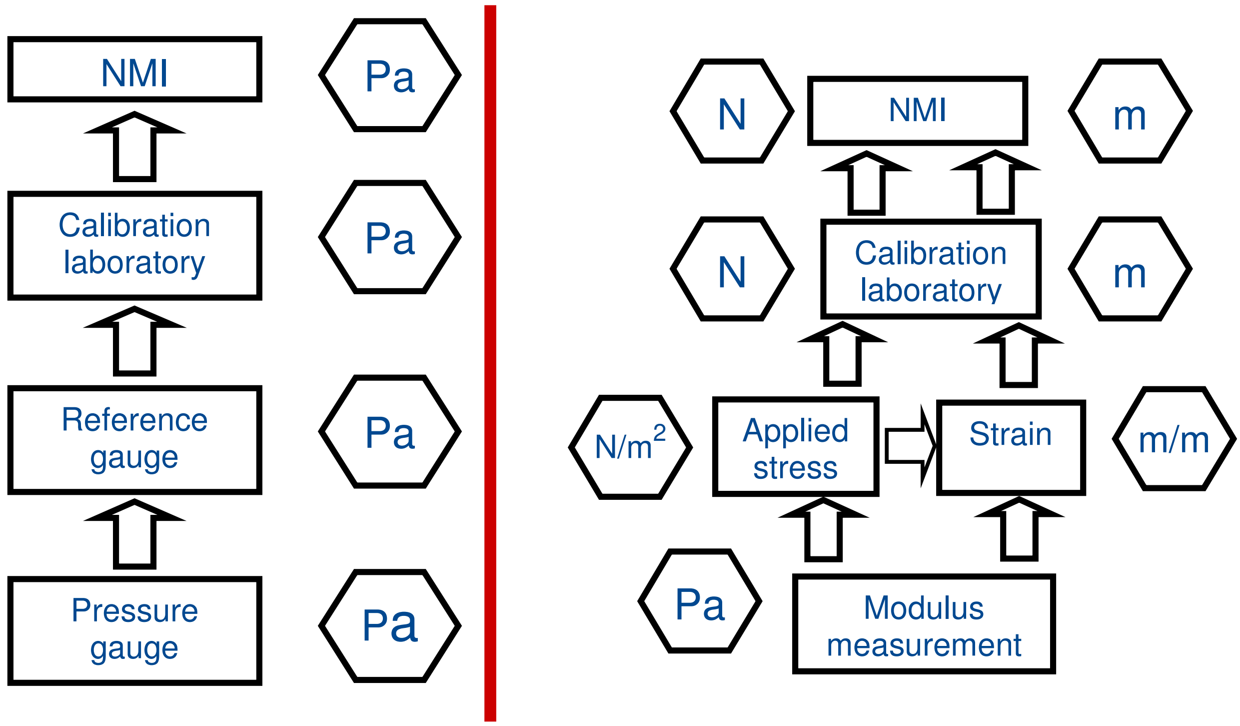


Figure 14.7 Comparison of the choice of dimensional units

15 DISCUSSION

15.1 WHAT ARE THE NEEDS – ECONOMIC AND REGULATORY DRIVERS?

The measurement of material properties has a major impact on the well-being of people, the security of national infrastructure and the effectiveness of world trade, as without accurate testing there cannot be any guarantee of the ability to deliver the specified products to meet all the individual needs. As noted previously, nearly all human endeavours involve the use of materials of one kind or another. Furthermore, from a cost-effectiveness viewpoint, the need for, and benefits from, a mutual recognition arrangement apply equally strongly to material property determination as to other fields of metrology.

Cases have been presented in section 6.1 where, for example, European Directives regarding products (including whole buildings) require critical values of material properties to be measured in order to demonstrate, through a traceability route, compliance with the applicable Directive. Unfortunately, it has also been demonstrated in section 14.1 that even a "simple" property that is frequently used in sophisticated FEA (Finite Element Analysis), such as the room temperature Young's Modulus of steel is not measured reliably with sufficient accuracy and repeatability.

The scatter of results and their uncertainty is equally important as their mean values, as in many cases design values (e.g. A and B values) are based on the relationship "Design value = (Mean – 2 Standard Deviations)", so that, high scatter can mean that the design value is so low as to be unusable. One NMI (NPL) has undertaken test work funded by a company to check the scatter in the results from a previous test laboratory rather than to check the mean values, the latter being as expected.

15.2 WHO ARE THE STAKEHOLDERS?

We could say that all of mankind are stakeholders as users of materials in their everyday lives, but the main stakeholders are the regulatory and accreditation bodies, together with the designers and engineers who work on our behalf to ensure product safety and reliability. It is clear that ILAC and the associated national accreditation bodies, for instance, require a well-founded system of reference materials in order to conduct its affairs and discharge its responsibilities through accreditation and proficiency testing.

The increasing importance to our economy and quality of life of non-traditional areas of measurement has been already recognised by CIPM in its forward-looking reviews. The latest report "Evolving Needs for Metrology in Trade, Industry and Society and the Role of the BIPM" [3] points to the need to take a broad approach to the subject of the measurement of materials properties in order to identify the metrological needs and the support which may be needed from NMIs.

15.3 WHAT ARE THE CCs DOING/PLANNING/ABLE TO DO?

Material properties are already covered by the work of several CCs, including in some cases through dedicated working groups. It is clear that there is a case for the formation of other working groups to address a wider range of properties. All these working groups would benefit from the involvement of experts with wider materials expertise, as available through VAMAS. The work of the CCM, CCAUV, CCEM and CCPR, for instance, has unsatisfied needs for reference materials with the required uniformity and stability (i.e. absence of ageing effects). It should be noted that material properties currently studied by the CC WGs do not always require the highest precision of the corresponding SI units (e.g. dielectric properties or thermal conductivity), but the work undertaken contributes to establishing higher order methods and reliable traceability for these properties.

15.4 WHAT ARE THE NMIs DOING/PLANNING/ABLE TO DO?

Many NMIs have encompassed materials measurement research for many years, and have been positive supporters of the original G7 VAMAS pre-normalisation initiative. It is noted that many countries outside the G7 are now keen to join VAMAS and are establishing materials research in their NMIs. Representatives of the NMIs in these countries participated actively in the work of this CIPM working group.

15.5 WHAT IS VAMAS DOING/ABLE TO DO? WHAT SHOULD IT DO?

VAMAS, although currently dedicated to improving the materials measurement infrastructure, could return to the approach followed at its inception of developing a strategic view of future needs (c.f. responding to bottom-up proposals) and establishing appropriate programmes of work. This is particularly appropriate as the VAMAS Steering Committee becomes more global with the addition of countries, such as Korea. However, any review must include input from all the stakeholders such as CIPM, ILAC and the standards organisations.

15.6 WHAT ARE THE MEASUREMENT ISSUES?

The main issues are that material properties data, in order to have maximum value and reliability and to be fit for purpose, should be measured,

- using calibrated equipment traceable to national standards for load, length etc.,
- using an agreed method, normally from a published standard, and
- using traceably characterised materials.

It is important to understand that variation in measured results will often reflect the material inhomogeneity as well as uncertainties associated with the test method or operator variability. The data presented in section 14.1 suggests that there is not currently the required level of confidence in the measured data, even for intrinsic properties.

There is generally an absence of precision data associated with material property test methods. In cases where validation and proficiency testing have been conducted using an agreed method/standard the evidence often shows poor agreement and unacceptable scatter. The inclusion of an uncertainty budget to accompany measured values is usually a requirement of accreditors rather than a specification in the standard being used. These difficulties are prevalent even in areas that materials experts would think less challenging as they do not involve aggressive environments, extreme temperatures, anisotropic materials or time dependence (c.f. modulus of steel at room temperature).

15.7 TRACEABILITY – SPECIFIC NEEDS

If material data are to perform their essential role in the economy and in support of quality of life, there is an urgent need to ensure that all the measurement results (e.g. load, displacement, dimensions, microstructure and composition) are traceable. In many cases researchers, especially in academic institutions, do not use calibrated equipment and do not include assessment of uncertainty in their measurements.

As noted above, many testing standards do not require statements of uncertainty and are generally characterised by an absence of typical precision and bias data. The precision data are vital in understanding the “fitness for purpose” of the method, particularly when using the method to defend a position regarding either a liability for failure or compliance with a purchase specification.

15.8 REFERENCE MATERIALS

Reference materials can clearly be seen to play an important role in the work of some existing CCs, in calibrations and in proficiency testing. A listing of approved, reliable reference materials would be of substantial benefit to all concerned through advertising their availability and thus, increasing their profile, such as the COMAR hosted by BAM. They would also be available for use in the validation of new measurement techniques and test methods as they are developed.

15.9 PROFICIENCY TESTING – ILAC

It is likely that further discussions with ILAC and their constituent members would identify additional issues regarding unsatisfactory scatter in different test methods. The data shown by way of examples in this report suggest an essentially unsatisfactory position exists, even for well-established tests. Certainly there has been anecdotal evidence of goods being turned away for failing to pass testing on importation although this Working Group has not been able to explore this issue comprehensively.

15.10 WHAT IS THE INFRASTRUCTURE NEEDED?

The activities of the JCTLM suggest the infrastructure requirements, which might be appropriate for material testing. A listing of reference materials would be relevant and valuable, with the same freedom for the traceability to be not necessarily to SI only, but to other sources if appropriate. A listing of reference methods would also be of value, although in this case there may be acceptable cross-references to existing standards.

At this stage, the Working Group is not recommending the establishment of a joint committee for materials metrology, in the belief that a great deal can be achieved through closer collaboration between CIPM and VAMAS and increased activity in some of the Consultative Committees. This should be kept under review, however, and the developments envisaged in this report may well lead to the eventual creation of an appropriate joint forum.

15.11 NANO-SCALE METROLOGY

The area of nano-scale measurements is all-pervasive, from material properties to dimensional aspects as well as localised electrical or temperature measurements. There is an increasing need to understand the impact of the local material structure and state in making these measurements. Any work on nano-scale materials metrology could be used to explore the interactions of CCs with materials expertise on a wider basis and might form part of a wider study on the requirements of metrology at the very small scale, where it is not only the traceability of materials properties which is in question. Establishing the traceability of measurements of mass, length, force and pressure on this scale is extremely challenging.

15.12 QUALITY SYSTEMS

A major recommendation of this report is the need to apply good metrology practice at all stages of the measurement process. For example, in current work on magnetic, thermal and dielectric properties, the greatest uncertainty is associated with the measurement of the size (area and thickness) of the sample under test, rather than the SI unit directly related to the property being investigated (e.g. temperature scale for heat conductivity). Equally, for measurement of Young's Modulus, for example, force and displacement are the principal measurements, but specimen dimensions and the full characterisation of the material being tested (composition, microstructure and form) are equally important. In addition, other requirements, such as the need for good practice in applying the load correctly (i.e. well-aligned) in order to create a uniform stress are critical.

16 RECOMMENDATIONS

16.1 TO CIPM

1 The Working Group recommends that the CIPM should sign a Cooperation Agreement with VAMAS in order to ensure an ongoing dialogue and actions with a view to identifying key traceability issues affecting the accuracy and repeatability of the measurement of materials properties.

2 The Working Group recommends that the CIPM should instigate a further review in 3 or 4 years time to evaluate the progress made and determine what further action, if any, is required.

16.2 TO CONSULTATIVE COMMITTEES

The Working Group recommends that CC working groups should be established to stimulate comparisons, establish measurement capabilities in NMIs and identify suitable certified reference materials with known uncertainties.

3 The Working Group recommends that the CCEM should establish a working group on electromagnetic properties of materials.

4 The Working Group recommends that the CCAUV should establish a working group on acoustic properties.

5 The Working Group recommends that the CCM and CCL should consider the case for a joint working group on mechanical properties of materials, with VAMAS representation.

6 The Working Group recommends that materials WGs established by CCs should encourage participation of all-important stakeholders, including ISO/IEC, ILAC and VAMAS.

16.3 TO NATIONAL METROLOGY INSTITUTES

7 The Working Group recommends that NMIs should support materials metrology in their work programmes in order to implement and disseminate best practice in the measurement of materials properties.

8 The Working Group recommends that NMIs should encourage their staff to participate actively in the work of joint BIPM/VAMAS materials working groups.

16.4 To VAMAS

9 The Working Group recommends that VAMAS Steering Committee should initiate a top-down review, with other stakeholders, to identify priority actions in selected areas and draw these to the attention of CIPM, ILAC and NMIs.

10 The Working Group recommends that VAMAS develop with CIPM appropriate pilot studies (see Annex A for examples).

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ANNEX A POTENTIAL PILOT STUDIES *(outline proposals)*

A1. MEASUREMENT OF MODULUS ROUND ROBIN

Introduction

In spite of the long history of testing metals, recent experience suggests that the measurement of modulus is not trivial and there can be large discrepancies in the values measured even from experienced practitioners. Currently the only standard available for measuring the Young's modulus of metals from the tensile test is ASTM E111, and there are many issues that contribute to the variability, which still need to be addressed. The European TENSTAND project reported on the accuracy of modulus measurement from the conventional tensile test, with recommendations for a separate procedure for modulus measurement, but this has not been progressed. Work has been carried out in the past on metal matrix composites, which has also highlighted strain measurement and data analysis as key factors affecting the quality and accuracy of the results.

Dynamic methods are sometimes used, but the tests are primarily those developed for the testing of ceramics.

There are also issues with the testing of modulus at temperature, where reliable data and test methods are not available.
Although there are no reference materials specifically developed for modulus measurement, a BCR Nimonic 75 tensile reference material does exist. This has not been fully certified for modulus; and the proposal within this exercise is to examine the tensile and dynamic modulus techniques with the aim of validating and certifying the material for modulus measurement.

Round-robin organisation

- Source BCR Nimonic tensile reference material and prepare test pieces.
- Circulate manufactured test pieces for tensile and dynamic measurement
- Specify test conditions – alignment, loading, strain measurement and data analysis
- Collate results and report

Report
- Report Young's Modulus

Time schedule

- 6-12 months

A2. GLASS TRANSITION (T_g) OF CURING RESIN SYSTEMS

Introduction

One example of materials metrology is the measurement of T_g the glass transition temperature, which is a materials property but reported as a temperature value, which is actually determined through a wide range of other measurement techniques, including:-
- ultrasonics,
- dielectrics,
- calorimetry (DSC),
- expansion,
- mechanical vibration (DMA),
- optical coherence tomography (OCT).

The value of Tg is often used as in materials qualification and specification requirements, as Tg is related to service temperature capability. See Standard Qualification Plan.

These above techniques are all used as secondary measures of degree of cure and shelf-life. Some of these techniques are better for off-line, rather than on-line measurements.

Standards exist for DSC (ISO 11357-3/ASTM E 1356), DMA (ISO 6721-11, ASTM D 3418). No standards for Tg by ultrasonic, dielectric, calorimetry and OCT.

NPL has a project to calibrate these techniques for material of different cure state against a "chemical" measure of cure (e.g. FTIR).

Round-robin activities

Supply panel of fully cured material

Glass-transition temperature to be determined by a range of techniques, as above.

Note NPL will supply a temperature reference specimen for calibration of DMA equipment.

Report

Report the glass-transition temperature

Time schedule

6-12 months

A3. MAGNETIC PROPERTY MEASUREMENTS

Introduction

An outline proposal for measuring magnetic properties is given below based on using the NPL reference core T1002. While the windings are identical, the left pair of secondary terminals (when viewing the core with the secondary terminals at the top) should be used.

To limit contributions due to temperature coefficients, the properties of the core should be measured for an ambient temperature of 20 ± 1 °C.

The laboratory should follow their own technical procedure for measuring the required properties.

Round-robin organisation

DC measurements

- Measure the normal magnetization curve and from this determine the maximum relative magnetic permeability.
- Determine the B versus H hysteresis curve up to a magnetic field strength of 2200 A/m.
- From the measured B versus H hysteresis curve determine the remanence, B_r, and the magnetic flux density coercivity, H_{cB}.
- The values determined should be submitted along with the estimated uncertainty in these values within one month of completing the measurements.

AC measurements

- Measurements to be made at a frequency of 50 Hz. For all measurements the secondary voltage waveform should be sinusoidal as defined in EN 60404 Parts 2 and 3.
- Determine the specific total loss, P_s, and the specific apparent power, S_s, at peak values of the magnetic flux density, B_{peak}, of 1.0 T, 1.1 T, 1.3 T, 1.5 T and 1.6 T.
- Determine the peak magnetic flux density, B_{peak}, for a peak magnetic field strength of 1000 A/m.
- The values determined should be submitted along with the estimated uncertainty in these values within one month of completing the measurements.

The following physical data can be assumed for the measurements:

Mass of core	= 0.5075 kg
Cross-sectional area of core	= 1.5030×10^{-4} m^2
Mean magnetic path length of core	= 0.44061 m
Number of primary turns	= 300
Number of secondary turns	= 150