SUMMARY

It was agreed at the 93rd Meeting of the CIPM that Professor Wallard (in collaboration with Dr Bennett) would respond to the letters from the Chairmen of VAMAS and ANMET, requesting further details about the perceived needs in the field of metrology for materials, and that Professor Wallard would also ask the Consultative Committees to define their activities and interests in the area. There has been little response from the Consultative Committees, but the request to VAMAS and ANMET resulted in a Workshop meeting at BIPM, Paris on 28th February 2005. This meeting confirmed the earlier recommendation that CIPM should create a Working Group and proposed an initial study of some clearly defined areas where there are significant metrology issues surrounding the variability of published measurement results and the associated regulatory requirements.
ABBREVIATIONS

ANMET - APEC Network for Materials Evaluation Technology
APEC - Asia-Pacific Economic Community
BAM - Bundesanstalt für Materialprüfung
BIPM - Bureau International des Poids et Mesures
CENAM - Centro Nacional de Metrología
CIPM - Comité International des Poids et Mesures
EIMRA - European Industrial Research and Management Association
ILAC - International Laboratory Accreditation Cooperation
KRISS - Korean Research Institute for Standards and Specifications
NPL - National Physical Laboratory
NMSD - National Measurement System Directorate
VAMAS - Versailles Project on Advanced Materials and Standards
1 BACKGROUND

The VAMAS Steering Committee meetings in 2003 and 2004 on “materials metrology” agreed that Dr Colin Lea (VAMAS Chairman until December 2004) would discuss with BIPM a route forward to strengthen the link between BIPM and VAMAS. This was undertaken in partnership with Dr Bahng (KRISS) - representing the APEC Network for Materials Evaluation Technology (ANMET). Following an ad hoc meeting at NPL in September 2004, presentations were made to the meeting of NMI Directors and the CIPM Meeting at the beginning of October. These presentations made a case on behalf of VAMAS and ANMET for a CIPM initiative in the field of metrology for materials, to build on work already undertaken by VAMAS and ANMET by using CIPM’s experience and reputation to provide international strategy, leadership and rigour.

Following the CIPM meeting, Andrew Wallard (BIPM) wrote to Consultative Committee Chairmen seeking their views, and Seton Bennett (CIPM Member) wrote to the ANMET and VAMAS Chairmen requesting further information about the present position of materials metrology and advice about the need for work under the auspices of CIPM. He also wrote to ILAC, requesting information about their perception of difficulties arising during the accreditation of materials testing laboratories.

Following correspondence with VAMAS and ANMET and further discussion with Professor Wallard, Dr Bennett convened a meeting of interested parties at BIPM on 28 February 2005, with a view to evaluating the options and making further, more detailed recommendations for CIPM action in response to identified needs in materials metrology.

2 ACTIVITIES

2.1 Ad-hoc Meeting at NPL – 15th September 2004

An ad-hoc meeting was held at NPL on 15 September 2004, hosted by Dr Lea. The aim of the meeting was to determine if there was sufficient interest and suitably robust arguments to support a proposal to CIPM for an increased role for “materials metrology”. The main presentations were given by Dr Lea and Dr Bahng, with several supporting shorter presentations. The meeting also reviewed a table of specific “tools” available to support the metrology of materials (Appendix A).

This meeting was attended by representatives from NMIJ, PTB, KRISS, INMETRO, CENAM, LNE, BIPM, BAM and NPL. There was general agreement that a case should be prepared for submission to CIPM at the October 2004 meeting. An outline skeleton of the case was assembled at the meeting, with input from all present and NPL and KRISS subsequently prepared a full case to be presented at Sèvres by Dr Seton Bennett (NPL’s Deputy Director and CIPM Member).

2.2 NMI Directors and CIPM meetings at BIPM – October 2004

Presentations developed following the September meeting at NPL were made by Dr Bennett and Dr Bahng to the National Metrology Institute Directors prior to last year’s annual CIPM meeting. Following discussion at the CIPM meeting, Dr Bennett asked VAMAS and ANMET to provide more detailed information justifying the need for CIPM activity. This information was required to inform a workshop hosted by Professor Wallard (Director, BIPM) in February 2005 in Paris. At the same time, Professor Wallard contacted existing CCs via their Chairmen, to determine their comments on the issues and request any relevant case studies regarding materials metrology needs.

In order to obtain further information and opinions, Dr Sims at NPL also sent requests to all VAMAS Steering Committee members and chairs of Technical Working Areas, to
the VAMAS UK shadow committee, and to selected NPL colleagues. NPL responses identified, in particular, the difficulties associated with dielectric measurements of materials and difficulties with the underlying philosophy of materials research. The Institute for Reference Materials and Measurements (IRMM) also made a useful input on the confusion arising from different fracture toughness standards. IRMM is the JRC laboratory of the EC with reference materials responsibilities, and from May 2005, the official EC representative to the VAMAS organisation.

### 2.3 ANMET Meeting - December 2004

Dr Bahng as Chairman of ANMET invited the VAMAS Chairman to present the role of VAMAS at their next meeting to be held at CENAM, Mexico in December 2004. Following receipt of the request from Dr Bennett noted above, there was full discussion of the inputs for the February 2005 BIPM workshop. Due to the timescale it was agreed that VAMAS and ANMET would cooperate through a virtual working group led by NPL and KRISS.

### 2.4 Meeting at BIPM on 28th February 2005

Professor Wallard hosted this meeting at BIPM, which was attended by the following NMI representatives:

- Seton Bennett, *NPL*
- Phillipe Charlet, *LNE*
- Masahiro Okaji, *NMII*
- Tetsuya Baba, *NMII*
- Gun Woong Bahng, *KRISS*
- Seong Jai Cho, *KRISS*
- João da Jornada, *Inmetro*
- Humberto Brandi, *Inmetro*
- Yoshito Mitani, *CENAM*
- Colin Lea, *NPL*
- Graham Sims, *NPL*
- Erich Santner, *BAM*
- Andrew Wallard, *BIPM*
- Bob Kaarls, *BIPM*
- Robert Wielgosz, *BIPM*

Following a number of presentations, discussion centred on the metrology and traceability issues raised by materials testing. As part of the submission by NPL, a draft remit for a Joint Committee on Traceability in Materials Metrology was considered, based on the remit of the Joint Committee on Traceability in Laboratory Medicine (JCTLM). The goal of the JCTLM is “to provide a worldwide platform to promote and give guidance on internationally recognized and accepted equivalence of measurements in laboratory medicine and traceability to appropriate measurement standards” and the committee is a joint activity of CIPM, ILAC (International Laboratory Accreditation Cooperation) and the International Federation for Clinical Chemistry and Laboratory Medicine (IFCC), with IFCC in the role that could be taken by VAMAS for
materials testing. A prime driver for this joint committee was the IVD European Directive - Directive 98/79/EC (1998) on in vitro diagnostic medical devices, which is absent for the material metrology case. However, many directives aimed at safe "products" and the safety of the citizen (e.g. Toys, Constructive Products, Personal Protective Equipment) include requirements for traceable material test procedures, reference and calibration materials. An early review of these Directives is needed to determine the extent to which they present materials measurement challenges. In addition, further contact should be made with national accreditation bodies, as well as with ILAC, who will have direct experience of the traceability issues through accreditation of test laboratories.

Among the many fields of materials metrology, the following were identified as presenting significant measurement challenges:

- structural measurements – grain size;
- dielectric properties;
- powders - particle size distribution (definition of characteristic parameters, applicability of these parameters, measurement procedures, pilot study (key comparison?), differences in values from different methods), surface properties, shape, density;
- agreed data for use in modeling;
- thermodynamic - conductivity, heat transfer, phase analysis, expansion, heat capacity, emissivity, diffusivity;
- density of porous materials;
- mechanical properties - strength, hardness, modulus, creep, toughness, impact;
- magnetic and optical properties;
- wear, friction, lubrication and corrosion;
- thin films – adhesion;
- nano-structured materials;
- multi-phased materials - composites, aerosols, gels.

Common issues which arise in these fields include:

- definitions of measurands;
- traceability requirements v harmonization of methods;
- availability of CRMs and reference methods;
- identification of reliable reference data and materials;
- inherent properties and procedural definitions (e.g. hardness);
- absence of uncertainty budgets in standards;
- shortage of reliable calibration services;
- need for proficiency testing;
- need for inter-comparisons at NMI level;
- educational need – guidance documents;
• need for improved appreciation of underpinning science and modelling of properties;
• absence of international agreement on priorities for metrology of materials;
• definition of quality control systems;
• support for certification and accreditation;
• demands from regulations and trade;
• environmental and healthcare (e.g. biocompatibility).

The meeting considered six possible courses of action:

• the formation of a Consultative Committee in CIPM;
• the formation of a Joint Committee for Traceability in Materials Metrology;
• the inclusion of more materials activities in current CCs;
• the formation of a CIPM Working Group;
• the encouragement of VAMAS and ANMET to redirect their activities;
• a decision to do nothing.

The arguments for and against these options are summarised in Appendix B. Following extensive discussion it was agreed that a proposal for a Working Group on Materials Metrology should be prepared for presentation by Dr Bennett at the 2005 CIPM meeting. The proposal should include examples from three areas to support the case for the immediate initiation of a programme of work.

3 TRACEABILITY IN MATERIALS METROLOGY: EXAMPLES

3.1 Fracture toughness of materials
Here results are normally expressed in J.m$^{-2}$ (=N.m$^{-1}$), with traceability to the kilogram and the metre. Discrepancies between specification standards are reinforced by the dependency of results on the method used and there is a need to understand the underlying science and establish reliable traceability in all cases. Properly formulated intercomparisons are required to investigate different test methods and validate changes which would contribute to closer agreement between results. These would in turn contribute to the revision of ISO standards with the incorporation of test methods giving a higher degree of agreement. A source of reference materials is also required to validate test methods and support accreditation.

3.2 Modulus measurements on metals
This field provides many examples of high scatter between results from different laboratories, preventing the optimised use of the latest high-performance alloys. Once again traceability is required to the metre and the kilogram, with little clarity about the way in which this is achieved. That this is so is evidenced by the insistence of all the Formula 1 racing teams that all modulus measurements must be made by a single reference laboratory (NPL) in order to ensure consistency. There is once again a pressing need for comparisons to evaluate performance and improve consistency on the basis of agreed traceability routes and common measurement protocols.

3.3 Nano-mechanical testing
This new field presents unique measurement challenges because the traceability issues common to the measurement of materials properties are exacerbated by the extremely small quantities of material involved. From apparently simple parameters
like particle size, to more complex measurements of nanonewton forces applied to materials and of wear properties when the quantities of material removed are expressed in nanograms, it is not clear how traceability of the results to the SI is to be assured. Once again, intercomparisons with clear protocols will lead to better assessment of NMI capabilities with the potential for improvements in specification standards and reduced uncertainties for testing laboratories.

3.4 Dielectric properties

This is an example of work which could be undertaken under the direction of an existing Consultative Committee (CCEM). Dielectric measurements vary more and good metrological practice is less common than in other areas of RF and microwave metrology, and there is a pressing need for education on traceability issues and the realistic estimation of uncertainty. Furthermore there are known to be deficiencies in reference materials and the travelling standards available for comparison exercises. These are associated with material instabilities and problems with contamination. Further work is required to select materials that will minimise these inconsistencies and to ensure the reliability and traceability of measurements in this important field.

4 CONCLUDING COMMENTS AND RECOMMENDATION

The clear majority view at the meetings reported above was that CIPM should once again be requested to initiate action to provide international leadership in metrology for materials. This paper includes neither a thorough review of metrology for materials nor a catalogue of traceability issues in the field. It does, however, include some examples of areas where inconsistencies appear to be connected with inadequate traceability and a shortage of reference materials. These do not, for the most part, fall squarely within the responsibility of existing Consultative Committees, although in some cases it may be appropriate for CCs to initiate relevant work as appropriate.

Of the options listed in Appendix B, there is widespread agreement that a new Consultative Committee would not be appropriate in the absence of an identified work programme. While some of the known issues fall within the scope of existing CCs, this is not completely the case and there is no single CC which could undertake the sort of broad study required.

In the event that CIPM decides that there is no need for activity under its auspices, VAMAS has already considered the possibility of taking a further initiative itself by seeking to extend its membership in order to obtain wider international agreement on the solutions required. As was pointed out at last year’s CIPM meeting, VAMAS and ANMET might expect to make faster progress working together than could be achieved in a CIPM Working Group. While this may, but need not, be true, it would lead to the development of an international forum for materials metrology which would overlap with the CIPM in some fields and might be seen to be in competition for resources. Insofar as there are issues concerning units, standards and traceability to address, the CIPM and the countries of the Metre Convention acting through BIPM should remain the sole international authority for metrology.

The remaining options are to establish a joint committee (possibly with ILAC and VAMAS) or to set up a working group as recommended last year. ILAC, in their response, have expressed their support for the proposal that BIPM should take a more proactive interest in this field, citing “specific (and difficult!) issues in relation to metrology (eg traceability, measurement uncertainty and reference materials etc), which could be tackled, perhaps on a case by case basis”. While they confirm their wish to contribute to any further work, they stress that resource limitations would make attendance at meetings difficult. This inability on the part of ILAC to participate actively in any work in this field would limit the effectiveness of a Joint Committee.
I therefore recommend that CIPM should set up a temporary Working Group for a period of two or three years under the chairmanship of a CIPM member. The Terms of Reference of the Working Group would be as follows:

- to identify the need for traceable measurements in the field of metrology for materials not currently covered by the Consultative Committees;
- to identify opportunities and mechanisms for working with Consultative Committees in areas of common interest;
- to establish, in the first instance, the user needs for activity in materials metrology;
- to define the specific objectives, initial activities, and long-term aims for an ongoing programme in metrology for materials;
- to establish a methodology for traceability in materials testing;
- to recommend activities required to underpin this methodology, such as the organisation of key comparisons and the development of appropriate reference materials;
- to undertake or initiate pilot exercises where these can be completed within the period of operation of the Working Group;
- to liaise closely with other interested organisations;
- to report its conclusions to the CIPM.

The VAMAS Committee should be invited to participate in the activities of this Working Group, as well as other individuals with specific specialist expertise.
APPENDIX A: IDENTIFIED MATERIALS METROLOGY TOOL AREAS

<table>
<thead>
<tr>
<th>Materials Area</th>
<th>Individual technique/tools area</th>
<th>Calibration/traceability</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Phys-Chem Properties</td>
<td>1. Thermodynamic data (Gibb’s energy, MT data, wettability, polarisability)</td>
<td>Reference materials</td>
</tr>
<tr>
<td></td>
<td>2. Thermal analysis (DSC, DMA, PVT) (Tg, reaction energies)</td>
<td>Temperature, Force / mass, Displacement / length</td>
</tr>
<tr>
<td></td>
<td>3. Electrochemistry, (Thermodynamics link with MTDATA, pH, mass transport, capacitance)</td>
<td>Voltage, current, standard Reference electrode</td>
</tr>
<tr>
<td>B. Microstructural Properties</td>
<td>1. Microstructure and topological characterisation (Optical, scanning, EDAX, WDX, EBSD, AFM)</td>
<td>Reference images / artefacts Microscope grids</td>
</tr>
<tr>
<td></td>
<td>2. Compositional information (Spectroscopic chemical analysis/surface analysis)</td>
<td>Reference materials</td>
</tr>
<tr>
<td></td>
<td>3. Non-destructive evaluations (Ultrasonics, X-ray, EPSI, thermography, Acoustic Emission)</td>
<td>Reference images,</td>
</tr>
<tr>
<td>C. Constitutive Properties</td>
<td>1. Fluid flow (Rheology, surface tension, wetting, density)</td>
<td>Force / mass, volume Displacement/length</td>
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<tr>
<td></td>
<td>2. Non-mechanical testing (electrical, dielectric, magnetic, optical, thermal expansion/conductivity, heat transfer)</td>
<td>Current, voltage, frequency Displacement/length</td>
</tr>
<tr>
<td></td>
<td>3. Mechanical testing (Non-contact strain, strength/modulus at different scale, nano-indenter)</td>
<td>Force/mass Displacement / length</td>
</tr>
<tr>
<td>D. Modelling</td>
<td>1. Predictive modelling (MT data, micromechanics, molecular modelling)</td>
<td>Reference data</td>
</tr>
<tr>
<td></td>
<td>2. Analytical and numerical modelling (FEA, CFD, CoDA) Model testing/comparison/validation</td>
<td>Reference constitutive equations and parameters.</td>
</tr>
<tr>
<td></td>
<td>3. Uncertainty budgeting</td>
<td>Uncertainties on constants / data</td>
</tr>
</tbody>
</table>
APPENDIX B: SIX OPTIONS FOR NEXT STEPS

1. A new CC [Consultative Committee for Materials Metrology of CIPM]
   Against - Breadth of expertise, funding needed, few NMIs have interest, identifying designated institutes, lack of national calibration facilities.
   For - Clear focus, use CIPM process, secretariat in place, NMIs can provide leadership, coordination/apply rigour of uncertainty, promotion of MRA to regulators.

2. JCTMM [Joint Committee on Traceability in Materials Metrology: Laboratory Medicine model]
   Against - difficulty of reaching agreement, lack of clear goals, needs resources.
   For - direct involvement of other stakeholders, ability to overview, tailor its own profile.

3. Strengthen current CCs [Use existing CCs]
   Against - need for a work programme, do not own the issue, not a uniform response, incomplete coverage, MM input not welcomed or seen as appropriate.
   For - existing experts in base SI units, less impact on BIPM resources.

4. WG [Working Group of CIPM]
   Against - less formal, not seen as authoritative.
   For - intermediate solution, easily implemented/less formal, can be easily stopped, previously provided a successful route for introduction of new areas.

5. Re-direct VAMAS/ANMET [use existing organisations]
   Against - perception of rejection, high level participation limited, duplication with CIPM.
   For - MoU with ISO/IEC, materials science expertise, some awareness of uncertainty and SI system.

6. Do nothing [Rely on market forces and evolution]
   Against - others will fill the position less well, poor representation of materials fraternity, lack of support to ILAC and materials testing.
   For - no effort needed.