

Establishment of a Traceability Chain in Metrology for Materials

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Abstract

Materials metrology is becoming an increasingly important international issue, with the worldwide efforts to realize a free trade agreement. The establishment of a traceability chain in materials metrology is, however, difficult because the measurement of materials properties is commonly procedure-dependent. This means that the establishment of a traceability chain in measurement of materials properties is not so straightforward as in physical measurement. In fact, three essential components, namely reference procedure, reference machine, and reference materials, are required to establish a traceability chain in materials metrology. In this paper, strategies to establish a traceability chain in metrology for materials are discussed to provide comparability amongst testing laboratories.

1. Characteristics of Metrology for Materials

Metrology is defined as the *Science of Measurement*.¹ This definition is clear for physical measurands such as the speed of light, Hall coefficient, triple point, etc. However, it is not so simple when it comes to materials properties. Materials properties depend on the composition and structure of materials. This means that materials properties can be traceable back to mole unit for composition and length unit for microstructure, respectively. Theoretically, it can be said that measurands in materials metrology are these two parameters. However, as it is impossible to predict cell function with information about molecular composition and structure, it is the same in materials metrology. In other words, relationship between materials properties and composition and structure is not well defined until this time and it would not be possible within foreseeable future.

Because of this fact, materials properties can be defined by the measuring procedure itself for the moment. Some examples are strength, toughness, fatigue, wear, corrosion, etc. This means that measurement results have different values depending on measurement methods, whereas measurands related to physical phenomena including

inherent materials properties are independent on the measurement procedures (although the accuracy or uncertainty of measurement results can be affected by them). Table 1 is a list of the parameters of materials properties.² They are categorized into two groups, inherent materials properties and procedural materials properties.

Table 1. Categories of materials properties

a) Examples of inherent materials properties

Category	Subcategory	Quantities
Mechanical	Elasticity	Young's modulus, shear modulus, bulk modulus, compressibility, Poisson's ratio, elasticity tensor, compliance tensor, sound velocity
Thermal	Capacity	Specific heat, Grüneisen 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

b) Examples of procedural materials properties

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 modulus, Weibull characteristic strength
Thermal	Stability	Flammability
Durability	Thermo-mechanical	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

2. Establishment of a traceability chain in metrology for materials

For procedural materials properties, it is essential to develop a reliable and dependable procedure, i.e., internationally agreed reference procedure. Defining reference procedure is the first step to establish a traceability chain in materials metrology since measurement results depend on the procedure. The next step is the development of a reference machine to realize this reference procedure. The most important concern for reference machine is that it should have lower or at least the same level of uncertainty compared to the best commercial equipment available. With these tools, it is possible to give a certified value on reference material. Certified Reference Material (CRM) with internationally agreed upon reference value and uncertainty should be used to calibrate a measurement system in accredited laboratories as well as industries to reduce scattering of data and to provide comparability of data. In this process, a traceability chain in materials metrology can be established through CRM.

NMIs' role is to provide CRM with a value and uncertainty based on internationally agreed reference procedure. Since international normative standards, providing a fitness-for-purpose accuracy, are sometimes adopted for ease and speed of measurement, it could not be used as a reference procedure in most cases. Actually a large scattering in measurement results among laboratories was reported by recent round robin tests.^{3,4} This fact was also clearly demonstrated in hardness key comparison that has been carried out by the Working Group on Hardness (WGH) of the Consultative Committee for Mass and Related Quantities (CCM).⁵ The WGH is now working on to reduce ambiguous parts in hardness measurement procedure.

It may be argued that calibration of measuring instruments in a measurement system such as load cell and extensometer of a tensile testing machine may provide a traceability chain to SI unit. Although calibration of measuring instruments is required, it is not enough to provide or secure the comparability in measurement results due to the procedural properties of materials.

Hardness measurement gives a good example. Although measuring instruments of a hardness machine could be traceable back to mass and length standards, there is no way to guarantee comparability even among NMIs because it is not possible to confirm whether NMIs are carrying out the test in accordance with the reference procedure. This is very serious problem since the measurement results of procedural properties depend

on the procedure itself. In order to secure the comparability in hardness measurement, inter-comparison amongst NMIs using hardness reference blocks is essential. Certified value of primary hardness block with a declared uncertainty can be obtained through inter-comparison amongst NMIs. This CRM with a stated uncertainty is used to calibrate hardness machine.

Because of this fact, the terminology, 'calibration', cannot be applicable to measuring instruments in a measurement system. It should be called as 'verification' confirming whether measuring instruments satisfy requirements or not. Calibration is defined as a set of operation to establish the relationship between values of quantities indicated by a measuring system and the corresponding values realized by standards. For hardness, 'standard' is the certified value of CRM. Therefore traceability is established through unbroken chain of comparisons with a stated value and uncertainty of CRM. Using CRM for calibration of a measurement system, comparability among accredited laboratories as well as industries can be guaranteed. Figure 1 shows a traceability chain with three essential elements for the establishment of standards in materials metrology.

Similar case can be found in metrology for chemistry, where the result of analysis is affected by measurement methods just as the procedural properties of materials. For example, Consultative Committee for Amount of Substance (CCQM) decided to adopt Harned cell as the primary method of pH measurement and its traceability chain is established by a CRM, buffer solution, whose value is given by the Harned cell. This is why the working groups under CCQM were named after a particular measurement method, while the names of the other CC working groups are based on the measurand itself.

Under these circumstances, the question narrows down who should give a reference value to the CRM with reliable uncertainty that is acceptable to the user. When we consider the huge amount of materials-related products in the world market, this issue of securing comparability becomes urgent to save resources and to improve the safety of structural materials. Unfortunately, there is no international focal point to develop CRMs for the establishment of a traceability chain in materials metrology except in a few cases such as hardness. To provide equivalence in materials properties measurement, worldwide cooperation amongst NMIs is a prerequisite to secure

comparability among accredited laboratories.

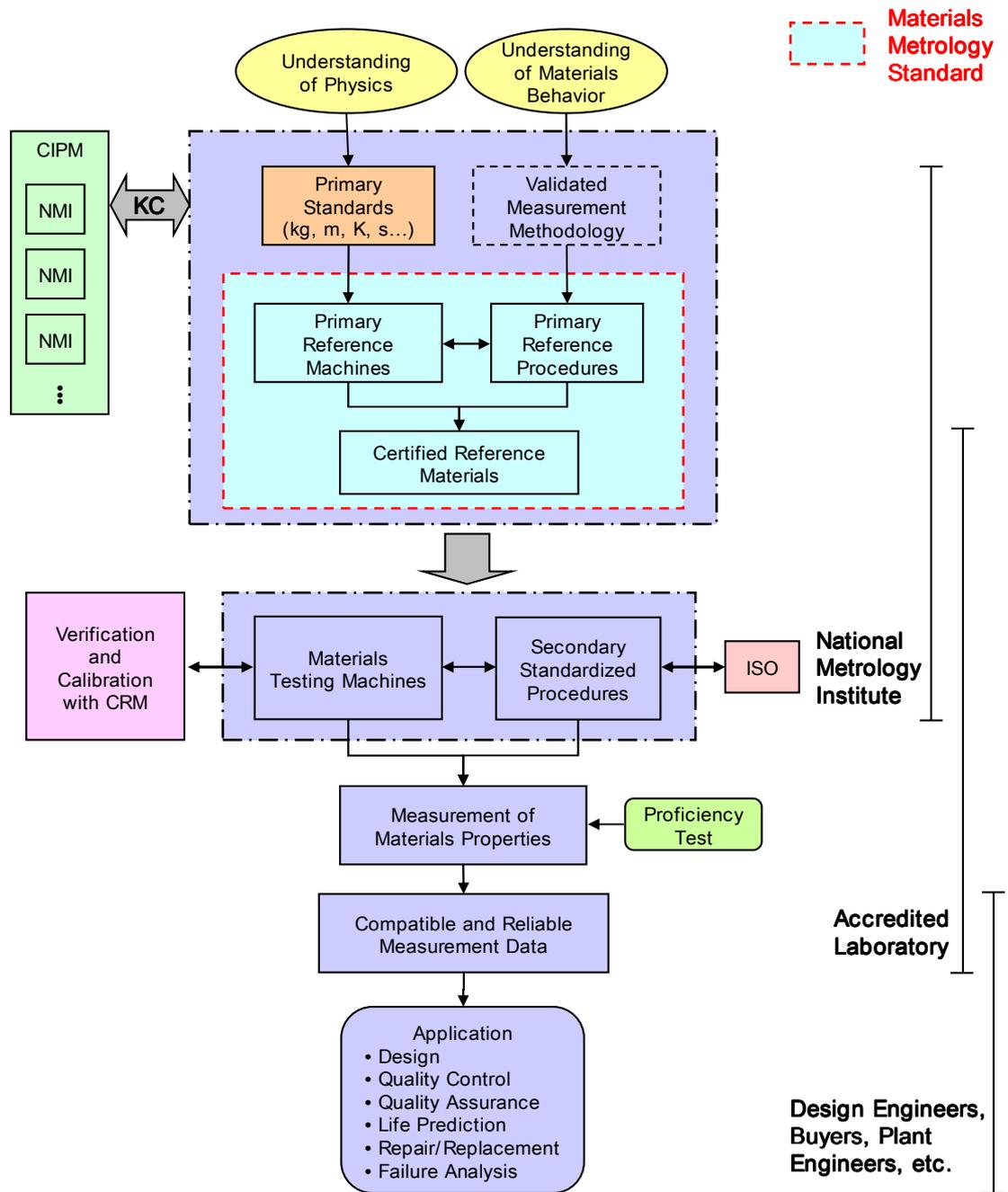


Fig. 1. Traceability chain in metrology for materials

3. Establishment of a Consultative Committee for Metrology of Materials (CCMM)

Looking at the current situation in materials metrology and considering the size of materials related industry, it seems that the resolution would be the establishment of a CCMM under the Committee International des Poids et Mesures (CIPM), dealing with matters on standards in materials metrology. The role of the CCMM will be the establishment of standards based upon reference procedure, reference machine, and reference materials for materials metrology through the organization of key comparisons and the development of CRMs. Considering the fact that Bureau International des Poids et Mesures (BIPM) has an authority in international measurement standards according to the Metre Convention, a CCMM under the CIPM will give an authority regarding the suggested works for the establishment of standards in materials metrology.

At the beginning, it would be proper to start with working groups on metrology for materials under the CIPM as was the case of CCQM. Initially, two possible working groups concerned with microstructure and mechanical properties of materials could be discussed by appropriate NMIs. The former is the area that is not covered by any working groups under CC. The latter is the area where the demand from industry is heaviest. The main roles of these working groups will be the development of reference procedure and CRMs, discussions about the requirements of reference machines, uncertainty evaluation, and key comparisons to provide comparability amongst NMIs or NMI-designated institutes in each area. The results of the activities might come out in a form as the Calibration and Measurement Capability (CMC) table in accordance with CIPM Mutual Recognition Arrangement (MRA).

4. Conclusion

Considering the great demand for materials metrology from industry as well as from accredited laboratories, it is timely and urgent to establish standards in materials metrology. Due to the unique characteristics of materials properties evaluation, especially as is seen in procedural properties, an international cooperation among NMIs is essential in order to develop reliable and compatible CRM, which is indispensable to improve the equivalency among accredited laboratories. At this time, available CRM in

this area is far less behind than the demand from industry as well as accredited laboratories.

To establish standards in materials metrology, three essential elements – reference procedure, reference machine, and reference materials – need to be developed and accepted. It is now the time to expand the activities of CIPM to establish standards in materials metrology. In this way, the establishment of a traceability chain in materials metrology would be possible.

¹ *International Vocabulary of Basic and General Terms in Metrology*, 2nd Ed., 1993.

² Ronald G. Munro, *Data Evaluation Theory and Practice for Materials Properties*, NIST Special Publication 960-11, 2003

³ M. K. Hossain and I. R. Sced, “Metrology for engineering materials”, *Materials Metrology and Standards for Structural Performance*, pp. 210-226, ed. B. F. Dyson et. al., Chapman & Hall, 1995.

⁴ Final Report of Proficiency Test TTM 2002 “Metallic Materials: Tensile Testing at Ambient Temperature,” Organized by IfEP (Institut für Eignungsprüfung), Dec. 2002

⁵ BIPM Key and Supplementary comparisons, Appendix B, CCM. H-S1a, Rockwell Hardness