Letter from EUROPE

This letter deals with **Measurement and Testing in Engineering**. It stems from reflections of this years' NCSLI Conference on *Metrology's Impact on Global Trade* and was caused by the discussion remark "Metrology is necessary but not sufficient" in engineering. The contribution illustrates the role of *Reference Procedures* and suggests to combine metrology and testing methodologies to obtain a general scheme for measurement and testing in engineering.

Challenges on measurement in engineering

Measurement is defined according to the International Vocabulary of Metrology (VIM) as *process of experimentally obtaining one or more quantity values that can be reasonably attributed to a quantity*. From the view of technology and engineering additional information on the "characteristics of the technical object", to which the measured numerical quantity values are related, are needed. The following example from the field of mechanical engineering may exemplify this statement.

For engineered products, important characteristics are for example *elasticity, hardness, strength,* etc. They describe the response of a technical object to external mechanical loading and are crucial for the reliability and safety of mechanical components.

- Metrologically, the quantities involved are force F (or torque) and the length measures l of the cross-sectional area A of the technical object.
- Technologically, the mechanical characteristics and their scatter depend on at least three basic groups of influencing parameters, illustrated by simple examples:
  1. *Intrinsic* properties of the object, like chemical composition, microstructure, etc.
     - metal hardness is different from polymer hardness.
  2. *Extrinsic* mechanical action: tension, compression, bending, shear, torsion
     - bending strength is different from shear strength.
  3. *Time-dependence* of the loading mode forces: static, dynamic, impact, stochastic
     - dynamic strength is different from static strength

The crucial point is to describe metrologically (1), (2), (3) and their "traceabilty".

Figure 1 illustrates the methodology of the determination of mechanical characteristics of technical objects and the stress-strain curve from which quantity values of the basic mechanical properties which are used in technology and engineering are derived.

An approach of a combined methodology for measurement and testing

A general scheme for measurement and testing can evolve if the fundamentals of metrology are combined with the methodology of testing. The scheme should be self-explaining as only terms defined in the VIM are used – see also the EURAMET brochure *Metrology in short*. (The sub-items of *Reference material* are explained in **me·trol'o·gist**, 2/3, 2009, p. 42 and those of *Reference procedure* in the following paragraph.)

The key of this scheme is the notion that metrologically determined measurands are quantity values which have to be related to the characteristics of technical objects determined by testing, supported by reference materials and reference procedures.

Template for reference procedures

Reference procedures are procedures of testing, measurement or analysis, thoroughly characterized and proven to be under control, intended for

- determination of reference values, or
- characterization of reference materials including reference objects, or
- quality assessment of other procedures for comparable tasks.

The uncertainty of the results of a measurement performed as part of a reference procedure must be adequately estimated and appropriate for the intended use.

When dealing with a range of measurement or test procedures, it is useful to have the most important characteristics presented in a standard format. A generic template for the presentation of reference procedures was developed in Germany at the Federal Institute for Materials Research and Testing (BAM) and utilised in their catalogue (www.bam.de). According to this template, reference measurement procedures are presented as follows:

- *Title* – the title of the procedure.
- *Key words* – key words for the procedure.
- *Quantities and items measured* – the quantities measured, the kind of objects which can be investigated and important measuring conditions.
- *Measuring range and Uncertainty of measurement* – the range of values, i.e. the working range as a reference procedure, for each quantity measured, and an associated uncertainty range. Uncertainty of measurement is an expanded uncertainty according to the GUM for an approximate confidence level of 0,95 (coverage factor k = 2), expressed as a relative uncertainty in % or as an absolute uncertainty in units of the quantity measured.

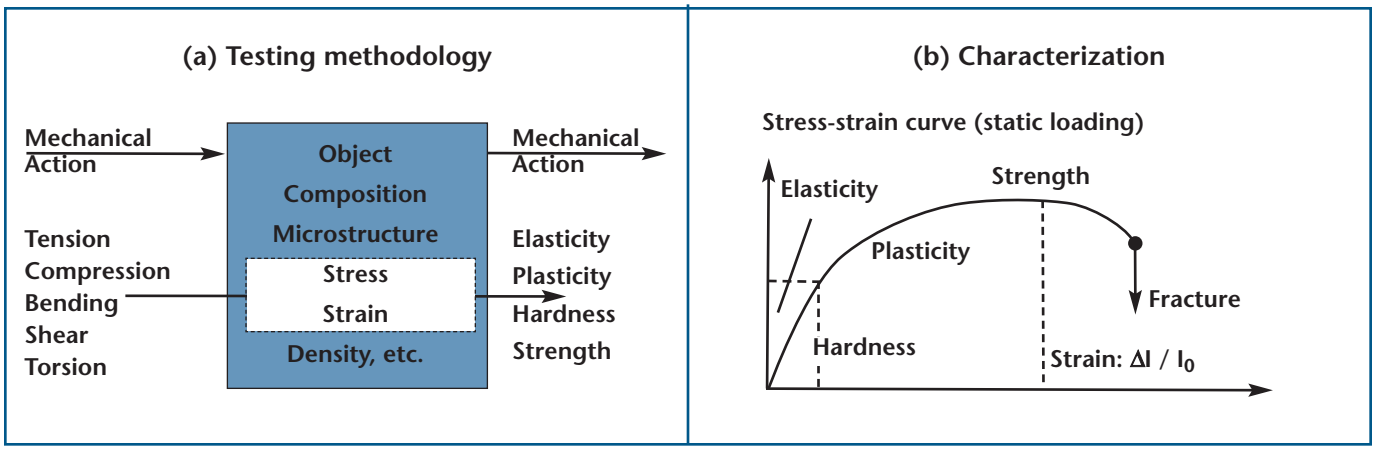

Figure 1. Determination of mechanical characteristics of technical objects. *Source: Springer Handbook of Materials Measurement Methods* (Editors, Czichos, Saito, Smith) 2006.

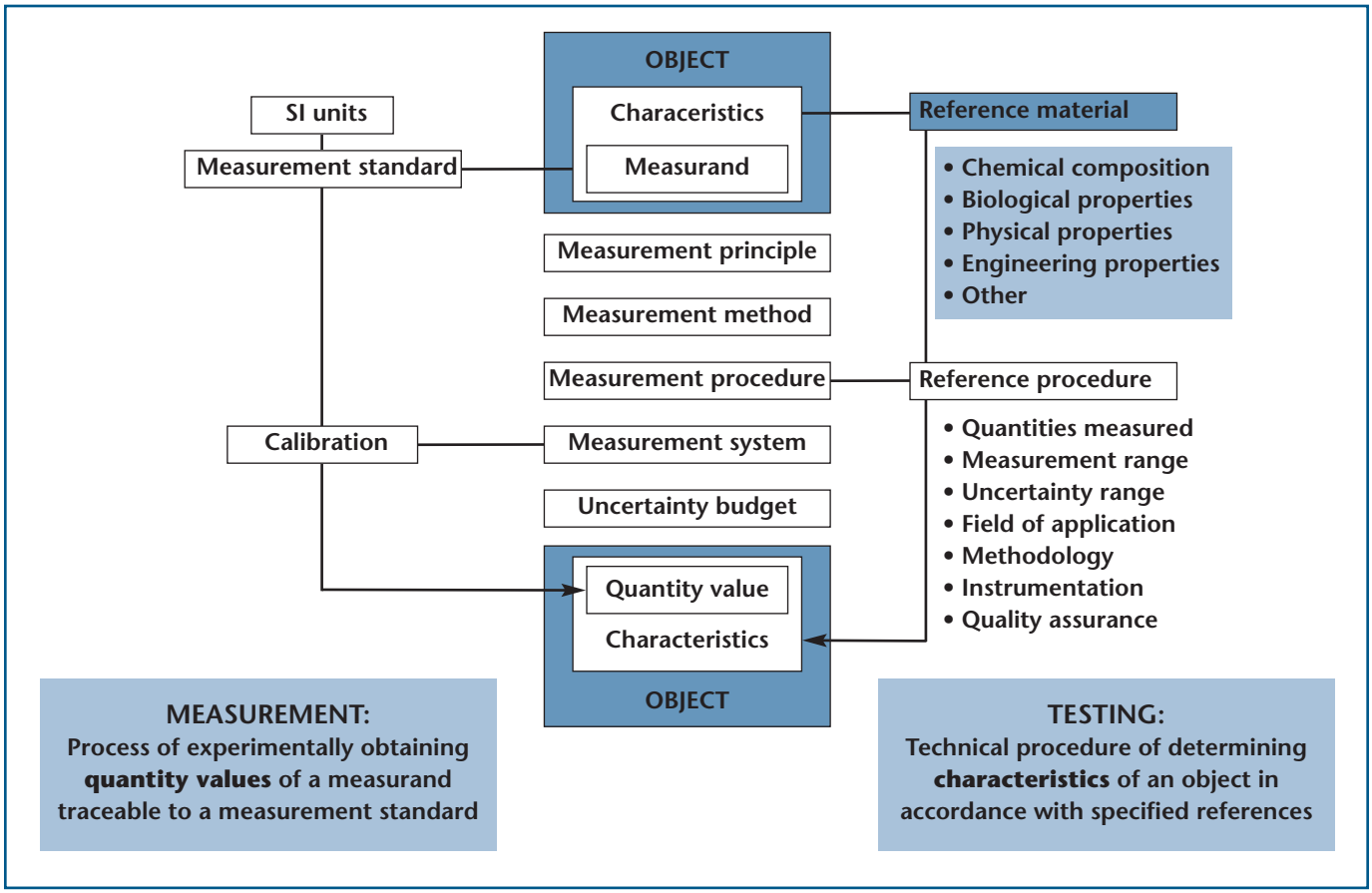

Figure 2. Measurement and testing in engineering: a general scheme.

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- *Fields of application* – the kind of tasks for which the procedure is currently in use, or may be utilised as a "reference": quality assessment of other procedures, characterisation of reference materials and objects, determination of reference values and other high-level tasks.
- *Methodology and instrumentation* – the method of measurement and essentials of the measuring system.
- *Qualification and quality assurance* – supporting evidence for the critical figures of merit, in particular measurement uncertainty (e.g. successful participation in inter-laboratory comparisons), and efforts taken for measurement quality control.
- *Further information* – free-style presentation of additional information, in particular including pictures, diagrams and references.

The system of reference procedures established by BAM comprises about 70 well-defined, quality-controlled RPs for:

1. Inorganic chemical analysis
2. Organic chemical analysis
3. Gas analysis and gas measurement
4. Microprobing and microstructural analysis
5. Testing of mechanical-technological properties
6. Testing of optical and electrical properties
7. Non-destructive testing,
8. Testing of surface and layer properties.

In the following some examples of the system of reference procedures are given:

In conclusion: Reference procedures apply to measurement, testing, and analysis, i.e. procedures to characterize materials and engineered components. They are used for determining reference values of materials characteristics, to validate products and procedures and to control material-related aspects of quality, reliability, and safety of products and technical systems. The combination of metrology and reference procedures is a crucial and sufficient methodology for measurement and testing in engineering.

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Characterisation of Materials Composition

Inorganic Materials: Analysis of chromium in steel					
Key words	Composition topic	Reference range	Uncertainty range	Methodology and instrumentation	Application examples
Highly alloyed steel, alloys, titimetry	Chromium content in steels and alloys	5 … 500 g/kg	0.02 … 2 g/kg	Titration with combined Pt-electrode/reference electrode; pure potassium titration solution	Certification of reference materials, standards for the iron and steel industry
Organic Materials: Analysis of pesticides in food					
Key words	Composition topic	Reference range	Uncertainty range	Methodology and instrumentation	Application examples
Food, DDT, pesticides, GC-IDMS, chromatography	Chloropesticides in edile oils and fats or vegetables	Amount of single chloropesticiede: 01 … 10 µg/g	1,5 … 3 %	Sample preparation and cleanup; GPC; measurement; GC-IDMS; calibration; gravimetry, certified purity; ^{13}C isotopes	Food control: analysis of opesticides (e.g. in oil or vegetables), environmental control samples

Characterisation of Materials Microstructure					
Microstructure Analysis: Morphology analysis of inorganic and organic compounds					
Key words	Composition topic	Reference range	Uncertainty range	Methodology and instrumentation	Application examples
Polymorphy, X-ray analysis, powder diffractometry	3D-structure; configuration, bond length/angles, intermolecular interactions	Crystalline mixtures consisting of several polymorphs	1 … 5 %, depending on number and crystalinity of polymorphs	X-ray structure and phase analysis: single crystal diffractometer, CCD detector, powder diffractometer; ORDER-DIS-ORDER theory	Validation of NMR, IR and Raman analysis (see 2.2), characterisation of reference materials, importance for pharmacy developments
Surface Analysis: Measurement of nanolayer thickness of oxidized silicon					
Key words	Composition topic	Reference range	Uncertainty range	Methodology and instrumentation	Application examples
Nanoscience, silicon, ESCA	Oxid overlayer thickness of oxidized silicon	1 … 10 nm	0.3 … 3 nm	ESCA measurement of Si 2p photoelectron intensities in the substrate and in the overlayer	Characterisation of materials used in the silicon technology and nanotechnology

Characterisation of Materials Properties					
Mechanical Properties: Determination of hardness and other plastic-elastic characteristics					
Key words	Composition topic	Reference range	Uncertainty range	Methodology and instrumentation	Application examples
Hardness, elasticity indentation test	Indentation depth Martens hardness HM	HM: 2 … 10^4 N/mm^2	1 … 1.5 %	Instrumented indentation test DIN EN ISO 14577 (Vickers pyramid)	Certified reference materials, round robin test, traceability of calibration
Optical Properties: Determination of optical and dielectric material constants					
Key words	Composition topic	Reference range	Uncertainty range	Methodology and instrumentation	Application examples
Materials surfaces, coatings, spectroscopic ellipsometry	Optical constants (n, k), dielectric constants, layer thickness h	Thickness h = 1 nm … 10 µm	0.5 … 1 %	Reflection with polarized light, light sources: white light, HeNe laser, synchrotron radiation (BESSY Berlin)	Development and certification of reference materials, standards for surface technology