

1. Introduction to Metrology and Testing

This chapter reviews the methodologies of measurement and testing. It gives an overview of metrology and presents the fundamentals of materials characterization as a basis for

1. Chemical and microstructural analysis
2. Materials properties measurement
3. Materials performance testing

which are treated in parts B, C, and D of the handbook.

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In science and engineering, objects of interest have to be characterized by measurement and testing. Measurement is the process of experimentally obtaining quantity values that can reasonably be attributed to a property of

a body or substance. Metrology is the science of measurement. Testing is the technical procedure consisting of the determination of characteristics of a given object or process, in accordance with a specified method [1.1].

1.1 Methodologies of Measurement and Testing

The methodologies of measurement and testing to determine characteristics of a given object are illustrated in a unified general scheme in Fig. 1.1, which is discussed in the next sections.

1.1.1 Measurement

Measurement begins with the definition of the measurand, the quantity intended to be measured. The specification of a measurand requires knowledge of the kind of quantity and a description of the object carrying the quantity. When the measurand is defined, it must be related to a measurement standard, the realization of the definition of the quantity to be measured. The measurement procedure is a detailed description

of a measurement according to a measurement principle and to a given measurement method. It is based on a measurement model, including any calculation to obtain a measurement result. The basic features of a measurement procedure are the following [1.1].

- Measurement principle: the phenomenon serving as a basis of a measurement
- Measurement method: a generic description of a logical organization of operations used in a measurement
- Measuring system: a set of one or more measuring instruments and often other devices, including any reagent and supply, assembled and adapted to give information used to generate measured quan-

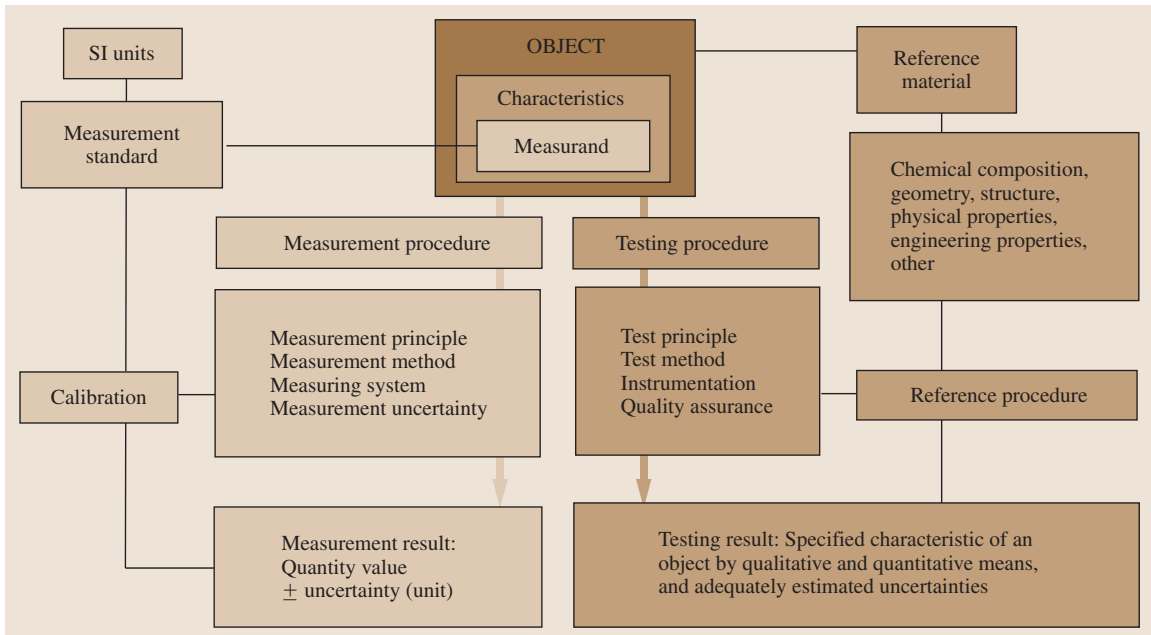


Fig. 1.1 The methodologies of measurement (light brown) and testing (dark brown) – a general scheme

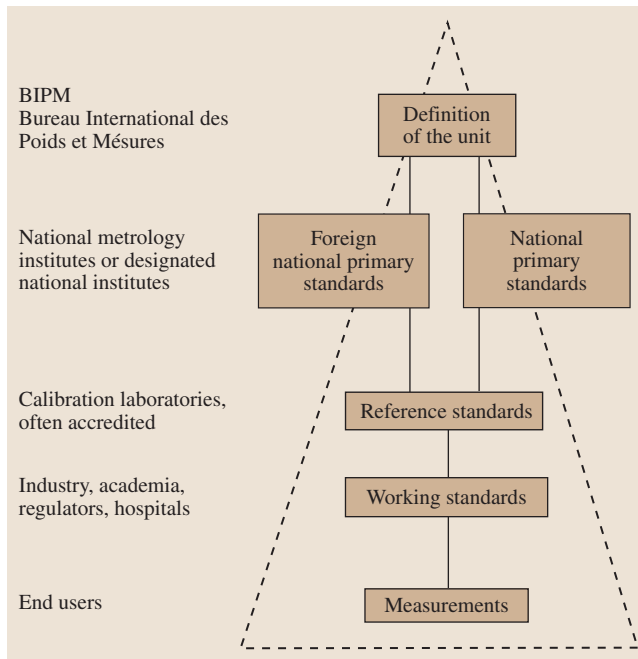


Fig. 1.2 The traceability chain for measurements

- **Measurement uncertainty:** a nonnegative parameter characterizing the dispersion of the quantity values being attributed to a measurand

The result of a measurement has to be expressed as a quantity value together with its uncertainty, including the unit of the measurand.

Traceability and Calibration

The measured quantity value must be related to a reference through a documented unbroken *traceability chain*. The *traceability of measurement* is described in detail in Sect. 3.2. Figure 1.2 illustrates this concept schematically.

The traceability chain ensures that a measurement result or the value of a standard is related to references at the higher levels, ending at the primary standard, based on the International System of Units (*le Système International d'Unités*, SI) (Sect. 1.2.3). An end user may obtain traceability to the highest international level either directly from a national metrology institute or from a secondary calibration laboratory, usually an accredited laboratory. As a result of various mutual recognition arrangements, internationally recognized traceability may be obtained from laboratories outside the user's own country. Metrological timelines in traceability, defined as changes, however slight, in

tity values within specified intervals for quantities of specified kinds

all instruments and standards over time, are discussed in [1.2].

A basic tool in ensuring the traceability of a measurement is either the calibration of a measuring instrument or system, or through the use of a reference material. Calibration determines the performance characteristics of an instrument or system before its use, while reference material calibrates the instrument or system at time of use. Calibration is usually achieved by means of a direct comparison against measurement standards or certified reference materials and is documented by a calibration certificate for the instrument.

The expression “traceability to the SI” means traceability of a measured quantity value to a unit of the International System of Units. This means metrological traceability to a *dematerialized reference*, because the SI units are conceptually based on *natural constants*, e.g., the speed of light for the unit of length. So, as already mentioned and shown in Fig. 1.1, the characterization of the measurand must be realized by a *measurement standard* (Sect. 1.2.4). If a measured quantity value is an attribute of a *materialized object* (e.g., a chemical substance, a material specimen or a manufactured product), also an object-related traceability (speciation) to a *materialized reference* (Fig. 1.1) is needed to characterize the object that bears the metrologically defined and measured quantity value.

Uncertainty of Measurements

Measurement uncertainty comprises, in general, many components and can be determined in different ways [1.3]. *The Statistical Evaluation of Results* is explained in detail in Sect. 3.3, and the *Accuracy and Uncertainty of Measurement* is comprehensively described in Sect. 3.4. A basic method to determine uncertainty of measurements is the *Guide to the expression of uncertainty in measurement* (GUM) [1.4], which is shared jointly by the Joint Committee for Guides in Metrology (JCGM) member organizations (BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP and OIML). The concept of the GUM can be briefly outlined as follows [1.5].

The GUM Uncertainty Philosophy.

- A measurement quantity X , whose value is not known exactly, is considered as a stochastic variable with a probability function.
- The result x of measurement is an estimate of the expectation value $E(X)$.

- The standard uncertainty $u(x)$ is equal to the square root of an estimate of the variance $V(X)$.
- *Type A uncertainty evaluation.* Expectation and variance are estimated by statistical processing of repeated measurements.
- *Type B uncertainty evaluation.* Expectation and variance are estimated by other methods than those used for type A evaluations. The most commonly used method is to assume a probability distribution, e.g., a rectangular distribution, based on experience or other information.

The GUM Method Based on the GUM Philosophy.

- Identify all important components of measurement uncertainty. There are many sources that can contribute to measurement uncertainty. Apply a model of the actual measurement process to identify the sources. Use measurement quantities in a mathematical model.
- Calculate the standard uncertainty of each component of measurement uncertainty. Each component of measurement uncertainty is expressed in terms of the standard uncertainty determined from either a type A or type B evaluation.
- Calculate the combined uncertainty u (the *uncertainty budget*). The combined uncertainty is calculated by combining the individual uncertainty components according to the law of propagation of uncertainty. In practice
 - for a sum or a difference of components, the combined uncertainty is calculated as the square root of the sum of the squared standard uncertainties of the components;
 - for a product or a quotient of components, the same *sum/difference* rule applies as for the relative standard uncertainties of the components.
- Calculate the expanded uncertainty U by multiplying the combined uncertainty with the coverage factor k .
- State the measurement result in the form $X = x \pm U$.

The methods to determine uncertainties are presented in detail in Sect. 3.4.

1.1.2 Testing

The aim of testing is to determine characteristics (attributes) of a given object and express them by qualitative and quantitative means, including adequately

estimated uncertainties, as outlined in the right-hand side of Fig. 1.1. For the testing methodology, metrology delivers the basis for the comparability of test results, e.g., by defining the units of measurement and the associated uncertainty of the measurement results. Essential tools supporting testing include reference materials, certified reference materials, and reference procedures.

- Reference material (RM) [1.6]: a material, sufficiently homogeneous and stable with regards to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties
- Certified reference material (CRM): a reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using a valid procedure
- Reference procedures [1.5]: procedures of testing, measurement or analysis, thoroughly characterized and proven to be under control, intended for
 - quality assessment of other procedures for comparable tasks, or
 - characterization of reference materials including reference objects, or
 - determination of reference values.

The uncertainty of the results of a reference procedure must be adequately estimated and appropriate for the intended use. Recommendations/guides for the de-

termination of uncertainties in different areas of testing include

- Guide for the estimation of measurement uncertainty in testing [1.7]
- Guide to the evaluation of measurement uncertainty for quantitative tests results [1.8]
- Guide for chemistry [1.9]
- Measurement uncertainty in environmental laboratories [1.10]
- Uncertainties in calibration and testing [1.11].

The methodology of testing combined with measurement is exemplified in Fig. 1.3 for the determination of mechanical characteristics of a technical object.

Generally speaking, the mechanical properties of materials characterize the response of a material sample to loading. The mechanical loading action on materials in engineering applications can basically be categorized as tension, compression, bending, shear or torsion, which may be static or dynamic. In addition, thermomechanical loading effects can occur. The testing of mechanical properties consists of measuring the mechanical loading stress (force/cross-sectional area $= F/A$) and the corresponding materials response (strain, elongation) and expressing this as a *stress-strain curve*. Its regimes and data points characterize the mechanical behavior of materials.

Consider for example *elasticity*, which is an important characteristic of all components of engineered structures. The elastic modulus (E) describes the rela-

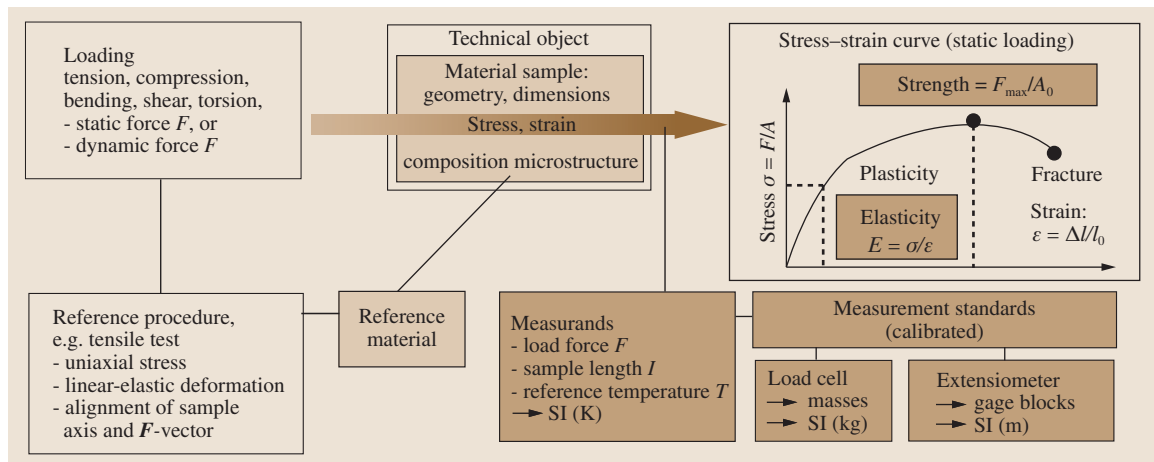


Fig. 1.3 The combination of measurement and testing to determine mechanical characteristics of the technical object

tion between a stress (σ) imposed on a material and the strain (ϵ) response of the material, or vice versa. The stimulus takes the form of an applied load, and the measured effect is the resultant displacement. The traceability of the stress is established through the use of a calibrated load cell and by measuring the specimen cross-sectional area with a calibrated micrometer, whereas the traceability of the strain is established by measuring the change in length of the originally measured gage length, usually with a calibrated strain gage. This, however, is not sufficient to ensure repeatable results unless a testing reference procedure, e.g., a standardized tensile test, is used on identically prepared specimens, backed up by a reference material.

Figure 1.3 illustrates the metrological and technological aspects.

- Metrologically, the measurands of the strength value are the force (F), area (A), and the length measurement (l) of the technical object, all at a reference temperature (T).
- Technologically and concerning testing, the mechanical characteristics expressed in a stress–strain curve depend on at least the following groups of influencing parameters, to be backed up by appropriate references.
 - The chemical and physical nature of the object: chemical composition, microstructure, and *structure–property relations* such as crystallographic shape-memory effects [1.12]; for example, strength values of metals are significantly influenced by alloying elements, grain size (fine/coarse), work-hardening treatment, etc.
 - The mechanical loading action and dependence on deformation amplitude: tension, compression, bending, shear, and torsion; for example, tensile strength is different from shear strength for a given material.
 - The time dependence of the loading mode forces (static, dynamic, impact, stochastic) and deviations from simple linear-elastic deformation (anelastic, viscoelastic or micro-viscoplastic deformation). Generally, the dynamic strength of a material is different from its static strength.

The combined measurement and testing methodologies, their operating parameters, and the traceability requirements are illustrated in a highly simplified scheme by the *confidence ring* [1.13] shown in Fig. 1.4.

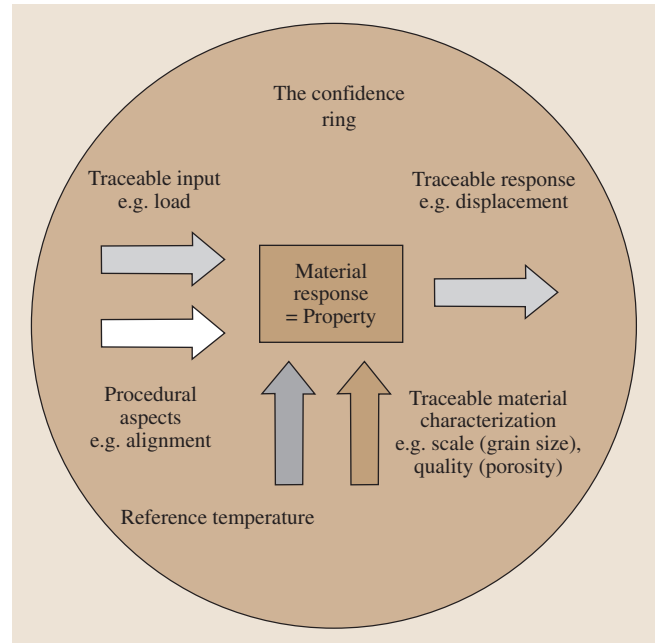


Fig. 1.4 Confidence ring for material property combined measurement and testing – note that separate traceability requirements apply to applied stimulus (load), response (displacement), and material characterization (grain size, porosity)

The confidence ring illustrates that, in measurement and testing, it is generally essential to establish reliable traceability for the applied stimulus and the resulting measured effect as well as for the measurements of any other quantities that may influence the final result. The final result may also be affected by the measurement procedure, by temperature, and by the state of the sample. It is important to understand that variation in measured results will often reflect material inhomogeneity as well as uncertainties associated with the test method or operator variability. All uncertainties should be taken into account in an *uncertainty budget*.

1.1.3 Conformity Assessment and Accreditation

In today's global market and world trade there is an increased need for *conformity assessment* to ensure that products and equipment meet specifications. The basis for conformity assessment are measurements together with methods of calibration, testing, inspection, and certification. The goal of conformity assessment

Table 1.1 Standards of conformity assessment tools

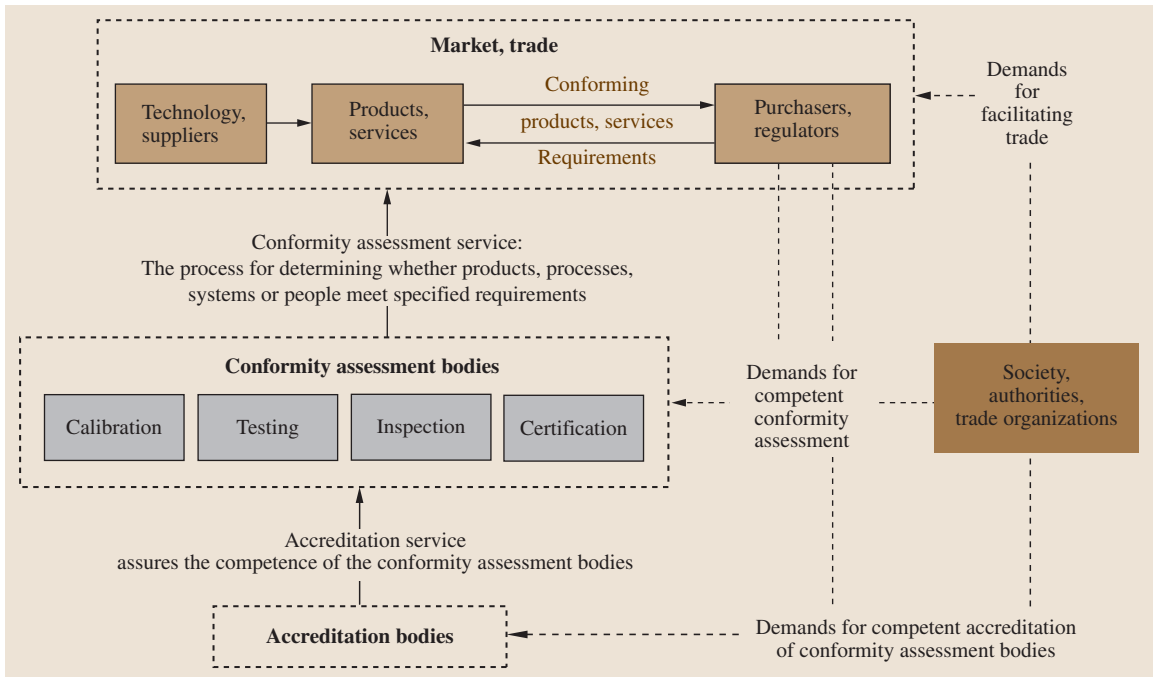
Tools for conformity assessment	First party Supplier, user	Second party Customers, trade associations, regulators	Third party Bodies independent from 1st and 2nd parties	ISO standards
Supplier's declaration	×			ISO/IEC 17050
Calibration, testing	×	×	×	ISO/IEC 17025
Inspection	×	×	×	ISO/IEC 17020
Certification			×	ISO 17021 ISO Guide 65

is to provide the user, purchaser or regulator with the necessary confidence that a product, service, process, system or person meets relevant requirements. The international standards relevant for conformity assessment services are provided by the ISO Committee on Conformity Assessment (CASCO). The conformity assessment tools are listed in Table 1.1, where their use by first parties (suppliers), second parties (customers, regulators, trade organizations), and third parties (bodies independent from both suppliers and customers) is indicated.

Along with the growing use of these conformity assessment tools there is the request for assurance of the competence of the conformity assessment bodies

(CABs). An increasingly applied and recognized tool for this assurance is *accreditation* of CABs.

The world's principal international forum for the development of laboratory accreditation practices and procedures is the International Laboratory Accreditation Cooperation (ILAC, <http://www.ilac.org/>). It promotes laboratory accreditation as a trade facilitation tool together with the recognition of competent calibration and testing facilities around the globe. ILAC started as a conference in 1977 and became a formal cooperation in 1996. In 2000, 36 ILAC members signed the ILAC Mutual Recognition Arrangement (MRA), and by 2008 the number of members of the ILAC MRA had risen to 60. Through the evaluation


Fig. 1.5 Interrelations between market, trade, conformity assessment, and accreditation

of the participating accreditation bodies, the international acceptance of test data and the elimination of technical barriers to trade are enhanced as recommended and in support of the World Trade Organization

(WTO) Technical Barriers to Trade agreement. An overview of the interrelations between market, trade, conformity assessment, and accreditation is shown in Fig. 1.5.

1.2 Overview of Metrology

Having considered the methodologies of measurement and testing, a short general overview of metrology is given, based on *Metrology – in short* [1.5], a brochure published by EURAMET to establish a common metrological frame of reference.

1.2.1 The Meter Convention

In the middle of the 19th century the need for a worldwide decimal metric system became very apparent, particularly during the first universal industrial exhibitions. In 1875, a diplomatic conference on the meter took place in Paris, at which 17 governments signed the diplomatic treaty *the Meter Convention*. The signatories

decided to create and finance a permanent scientific institute: the *Bureau International des Poids et Mesures* (BIPM). The Meter Convention, slightly modified in 1921, remains the basis of all international agreement on units of measurement. Figure 1.6 provides a brief overview of the Meter Convention Organization (details are described in Chap. 2).

1.2.2 Categories of Metrology

Metrology covers three main areas of activities [1.5].

1. The definition of internationally accepted units of measurement

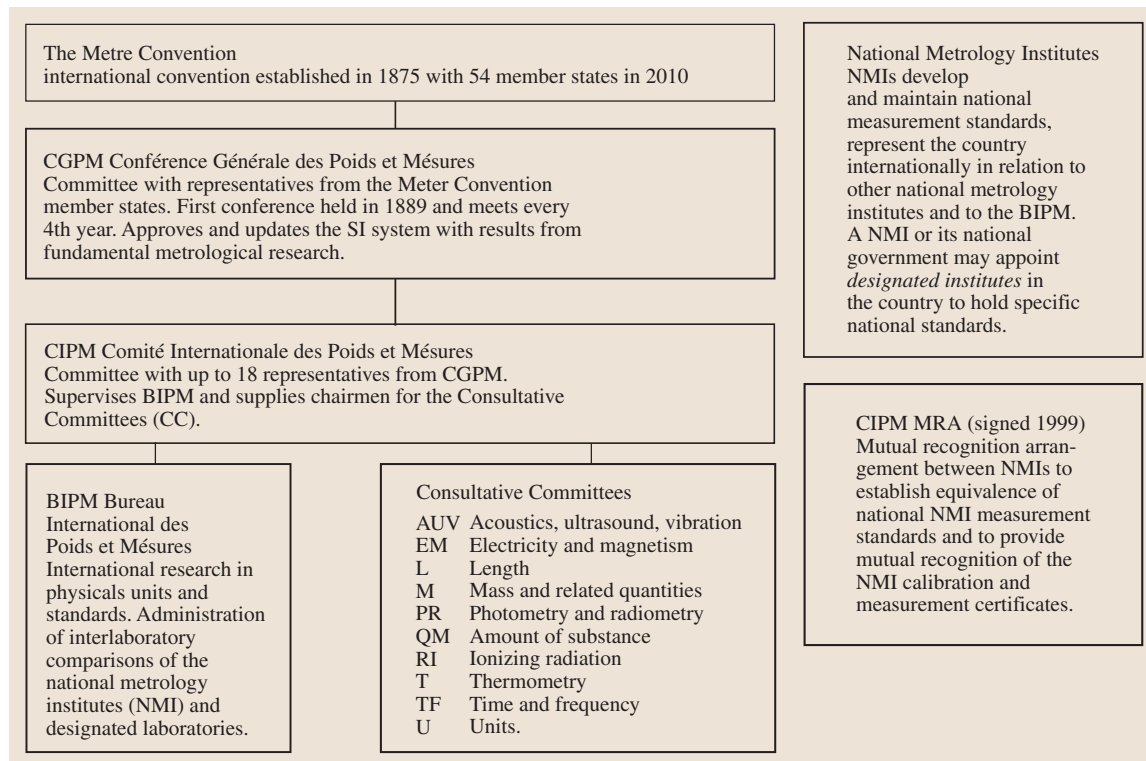


Fig. 1.6 The organizations and their relationships associated with the Meter Convention

2. The realization of units of measurement by scientific methods
3. The establishment of traceability chains by determining and documenting the value and accuracy of a measurement and disseminating that knowledge

Metrology is separated into three categories with different levels of complexity and accuracy (for details, see Chaps. 2 and 3).

Scientific Metrology

Scientific metrology deals with the organization and development of measurement standards and their maintenance. Fundamental metrology has no international definition, but it generally signifies the highest level of accuracy within a given field. Fundamental metrology may therefore be described as the top-level branch of scientific metrology.

Scientific metrology is categorized by BIPM into nine technical subject fields with different branches. The metrological calibration and measurement capabilities (CMCs) of the national metrology institutes (NIMs) and the designated institutes (DIs) are compiled together with *key comparisons* in the BIPM key comparison database (KCDB, <http://kcdb.bipm.org/>). All CMCs have undergone a process of peer evaluation by NMI experts under the supervision of the regional metrology organizations (RMOs). Table 1.2 shows the scientific metrology fields and their branches together with the number of registered calibration and measurement capabilities (CMCs) of the NMIs in 2010.

Industrial Metrology

Industrial metrology has to ensure the adequate functioning of measurement instruments used in industrial production and in testing processes. Systematic measurement with known degrees of uncertainty is one of the foundations of industrial quality control. Generally speaking, in most modern industries the costs bound up in taking measurements constitute 10–15% of production costs.

However, good measurements can significantly increase the value, effectiveness, and quality of a product. Thus, metrological activities, including calibration, testing, and measurements, are valuable inputs to ensure the quality of most industrial processes and quality of life related activities and processes. This includes the need to demonstrate traceability to international standards, which is becoming just as important as the measurement itself. Recognition of metrological competence at each level of the traceability chain can be established through mutual recognition agreements or arrangements, as well as through accreditation and peer review.

Legal Metrology

Legal metrology originated from the need to ensure fair trade, specifically in the area of weights and measures. The main objective of legal metrology is to assure citizens of correct measurement results when used in official and commercial transactions. Legally controlled instruments should guarantee correct measurement results throughout the whole period of use under working conditions, within given permissible errors.

Table 1.2 Metrology areas and their branches, together with the numbers of metrological calibration and measurement capabilities (CMCs) of the national metrology institutes and designated institutes in the BIPM KCDB as of 2010

Metrology area	Branch	CMCs
Acoustics, ultrasound, vibrations	Sound in air; sound in water; vibration	955
Electricity and magnetism	DC voltage, current, and resistance; impedance up to the megahertz range; AC voltage, current, and power; high voltage and current; other DC and low-frequency measurements; electric and magnetic fields; radiofrequency measurements	6586
Length	Laser; dimensional metrology	1164
Mass and related quantities	Mass; density; pressure; force; torque, viscosity, hardness and gravity; fluid flow	2609
Photometry and radiometry	Photometry; properties of detectors and sources; spectral properties; color; fiber optics	1044
Amount of substance	List of 16 amount-of-substance categories	4558
Ionizing radiation	Dosimetry; radioactivity; neutron measurements	3983
Thermometry	Temperature; humidity; thermophysical quantities	1393
Time and frequency	Time scale difference; frequency; time interval	586

For example, in Europe, the marketing and usage of the following measuring instruments are regulated by the European Union (EU) measuring instruments directive (MID 2004/22/EC)

1. Water meters
2. Gas meters
3. Electrical energy meters and measurement transformers
4. Heat meters
5. Measuring systems for liquids other than water
6. Weighing instruments
7. Taximeters
8. Material measures
9. Dimensional measuring systems
10. Exhaust gas analyzers

Member states of the European Union have the option to decide which of the instrument types they wish to regulate.

The International Organization of Legal Metrology (OIML) is an intergovernmental treaty organization established in 1955 on the basis of a convention, which was modified in 1968. In the year 2010, OIML was composed of 57 member countries and an additional 58 (corresponding) member countries that joined the OIML (<http://www.oiml.org/>) as observers. The purpose of OIML is to promote global harmonization of legal metrology procedures. The OIML has developed a worldwide technical structure that provides its members with metrological guidelines for the elaboration of national and regional requirements concerning the

manufacture and use of measuring instruments for legal metrology applications.

1.2.3 Metrological Units

The idea behind the metric system – a system of units based on the meter and the kilogram – arose during the French Revolution when two platinum artefact reference standards for the meter and the kilogram were constructed and deposited in the French National Archives in Paris in 1799 – later to be known as the Meter of the Archives and the Kilogram of the Archives. The French Academy of Science was commissioned by the National Assembly to design a new system of units for use throughout the world, and in 1946 the MKSA system (meter, kilogram, second, ampere) was accepted by the Meter Convention countries. The MKSA was extended in 1954 to include the kelvin and candela. The system then assumed the name the International System of Units (*Le Système International d'Unités*, SI). The SI system was established in 1960 by the 11th General Conference on Weights and Measures (CGPM): *The International System of Units (SI) is the coherent system of units adopted and recommended by the CGPM.*

At the 14th CGPM in 1971 the SI was again extended by the addition of the mole as base unit for amount of substance. The SI system is now comprised of seven base units, which together with derived units make up a coherent system of units [1.5], as shown in Table 1.3.

Table 1.3 The SI base units

Quantity	Base unit	Symbol	Definition
Length	Meter	m	The meter is the length of the path traveled by light in a vacuum during a time interval of $1/299\,792\,458$ of a second
Mass	Kilogram	kg	The kilogram is equal to the mass of the international prototype of the kilogram
Time	Second	s	The second is the duration of 9 192 631 770 periods of the radiation corresponding to the transition between the two hyperfine levels of the ground state of the cesium-133 atom
Electric current	Ampere	A	The ampere is that constant current which, if maintained in two straight parallel conductors of infinite length, of negligible circular cross-section, and placed one meter apart in vacuum, would produce between these conductors a force equal to 2×10^{-7} newtons per meter of length
Temperature	Kelvin	K	The kelvin is the fraction $1/273.16$ of the thermodynamic temperature of the triple point of water
Amount of substance	Mole	mol	The mole is the amount of substance of a system that contains as many elementary entities as there are atoms in 0.012 kg of carbon-12. When the mole is used, the elementary entities must be specified and may be atoms, molecules, ions, electrons, other particles, or specified groups of such particles
Luminous intensity	Candela	cd	The candela is the luminous intensity in a given direction of a source that emits monochromatic radiation of frequency 540×10^{12} Hz and has a radiant intensity in that direction of $1/683$ W per steradian

Table 1.4 Examples of SI derived units expressed in SI base units

Derived quantity	SI derived unit special name	Symbol	In SI units	In SI base units
Force	Newton	N		m kg s^{-2}
Pressure, stress	Pascal	Pa	N/m^2	$\text{m}^{-1} \text{kg s}^{-2}$
Energy, work, quantity of heat	Joule	J	N m	$\text{m}^2 \text{kg s}^{-2}$
Power	Watt	W	J/s	$\text{m}^2 \text{kg s}^{-3}$
Electric charge	Coulomb	C		s A
Electromotive force	Volt	V		$\text{m}^2 \text{kg s}^{-3} \text{A}^{-1}$
Electric capacitance	Farad	F	C/V	$\text{m}^{-2} \text{kg}^{-1} \text{s}^4 \text{A}^2$
Electric resistance	Ohm	Ω	V/A	$\text{m}^2 \text{kg s}^{-3} \text{A}^{-2}$
Electric conductance	Siemens	S	A/V	$\text{m}^{-2} \text{kg}^{-1} \text{s}^3 \text{A}^2$

SI derived units are derived from the SI base units in accordance with the physical connection between the quantities. Some derived units, with examples from mechanical engineering and electrical engineering, are compiled in Table 1.4.

1.2.4 Measurement Standards

In the introductory explanation of the methodology of measurement, two essential aspects were pointed out.

1. Measurement begins with the definition of the measurand.

2. When the measurand is defined, it must be related to a measurement standard.

A measurement standard, or etalon, is the realization of the definition of a given quantity, with stated quantity value and associated measurement uncertainty, used as a reference. The realization may be provided by a material measure, measuring instrument, reference material or measuring system.

Typical measurement standards for subfields of metrology are shown in Fig. 1.7 in connection with the scheme of the measurement methodology (left-hand side of Fig. 1.1). Consider, for example, dimensional metrology. The meter is defined as the length of the path

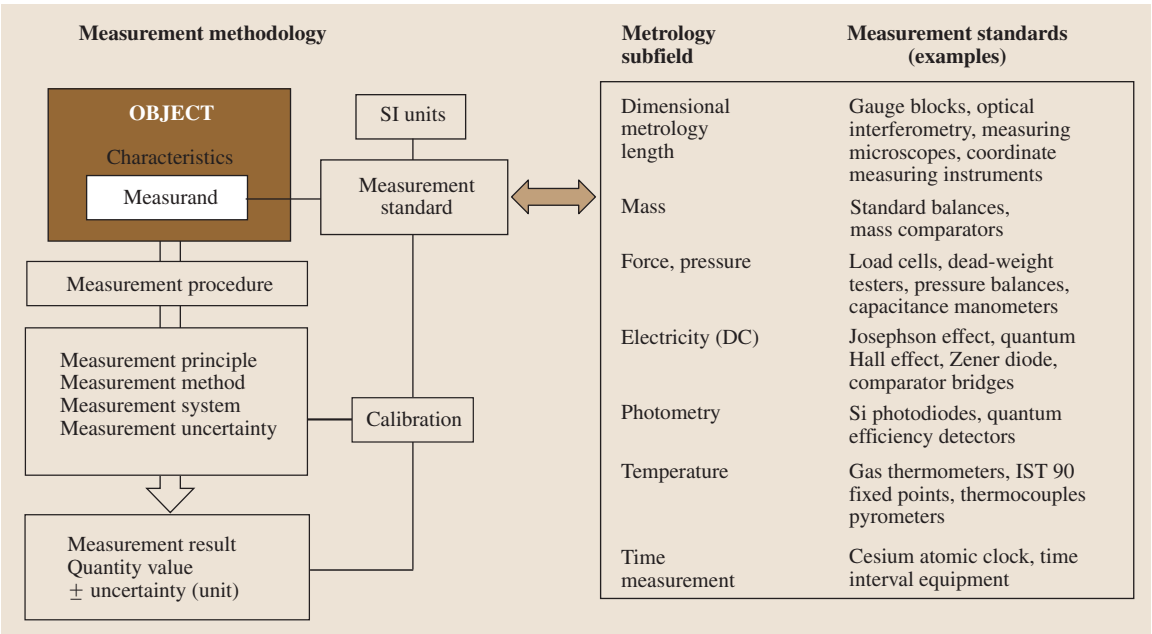


Fig. 1.7 Measurement standards as an integral part of the measurement methodology

traveled by light in vacuum during a time interval of $1/299\,792\,458$ of a second. The meter is realized at the primary level (SI units) in terms of the wavelength from an iodine-stabilized helium-neon laser. On sublevels, material measures such as gage blocks are used, and traceability is ensured by using optical interferometry to determine the length of the gage blocks with reference to the above-mentioned laser light wavelength.

A national measurement standard is recognized by a national authority to serve in a state or economy as the basis for assigning quantity values to other measurement standards for the kind of quantity concerned. An international measurement standard is recognized by signatories to an international agreement and intended to serve worldwide, e.g., the international prototype of the kilogram.

1.3 Fundamentals of Materials Characterization

Materials characterization methods have a wide scope and impact for science, technology, economy, and society, as materials comprise all natural and synthetic substances and constitute the physical matter of engineered and manufactured products.

For materials there is a comprehensive spectrum of *materials measurands*. This is due to the broad variety of metallic, inorganic, organic, and composite materials, their different chemical and physical nature, and the manifold attributes which are related to materials with respect to composition, microstructure, scale, synthesis, physical and electrical properties, and applications. Some of these attributes can be expressed in a metrological sense as numbers, such as density; some are Boolean, such as the ability to be recycled or not; some, such as resistance to corrosion, may be expressed as a ranking (poor, adequate, good, for instance); and some can only be captured in text and images [1.14]. As background for materials characterization methods, which are treated in parts B, C, D of the handbook, namely

- Chemical and microstructural analysis
- Materials properties measurement
- Materials performance testing

the essential features of materials are outlined in the next sections [1.15].

1.3.1 Nature of Materials

Materials can be natural (biological) in origin or synthetically processed and manufactured. According to their chemical nature, they are broadly grouped traditionally into inorganic and organic materials.

The physical structure of materials can be crystalline or amorphous, as well as mixtures of both structures. Composites are combinations of materials assembled together to obtain properties superior to those of their single constituents. Composites (C) are

classified according to the nature of their matrix: metal (MM), ceramic (CM) or polymer (PM) matrix composites, often designated as MMCs, CMCs, and PMCs, respectively. Figure 1.8 illustrates, with characteristic examples, the spectrum of materials between the categories natural, synthetic, inorganic, and organic.

From the view of *materials science*, the fundamental features of a solid material are as listed below.

- Material's atomic nature: the atomic elements of the Periodic Table which constitute the chemical composition of a material
- Material's atomic bonding: the type of cohesive electronic interactions between the atoms (or molecules) in a material, empirically categorized into the following basic classes.
 - Ionic bonds form between chemical elements with very different electron negativity (tendency

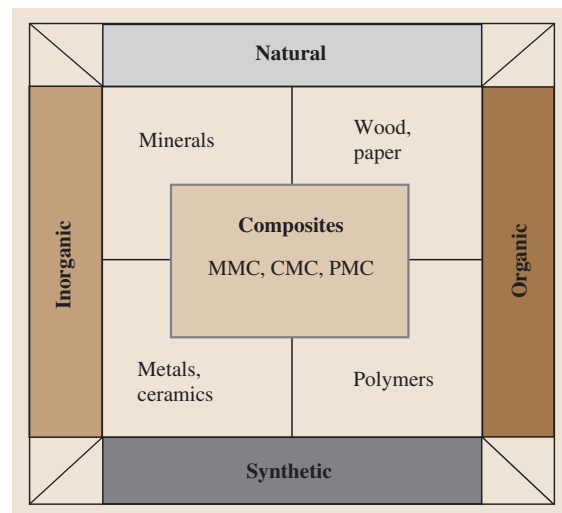


Fig. 1.8 Classification of materials

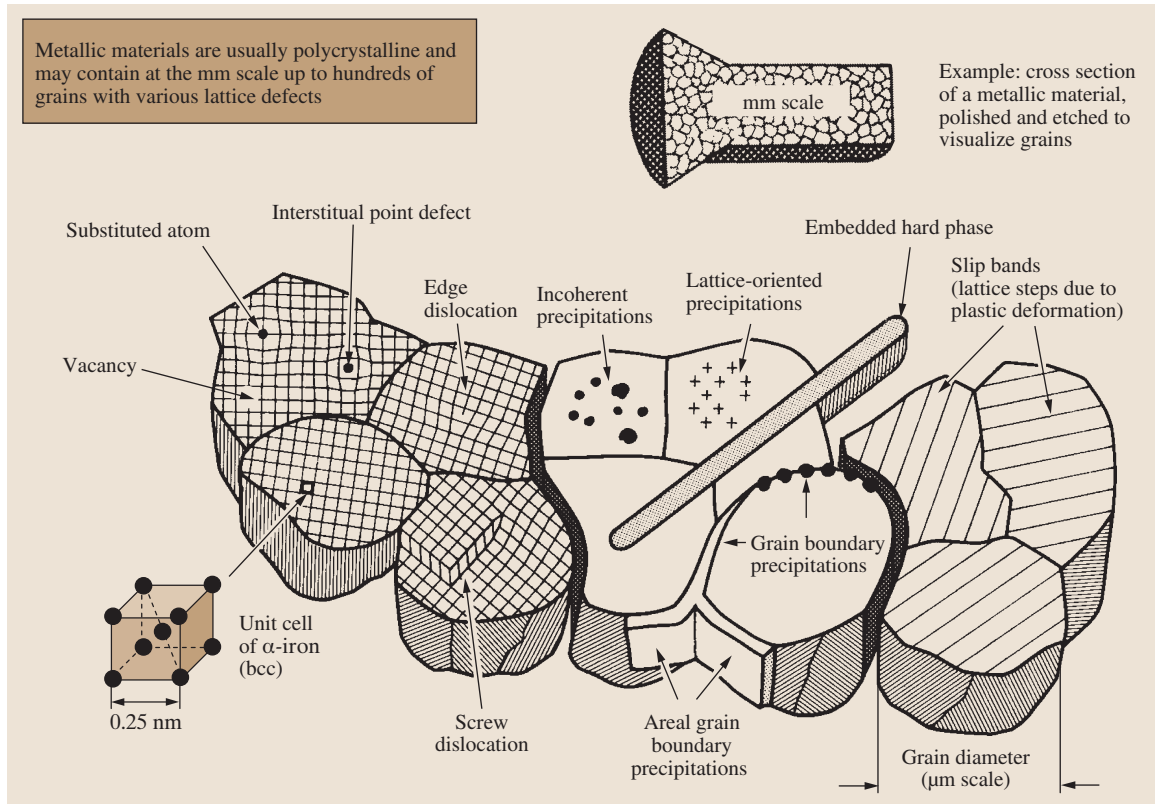


Fig. 1.9 Schematic overview on the microstructural features of metallic materials and alloys

to gain electrons), resulting in electron transfer and the formation of anions and cations. Bonding occurs through electrostatic forces between the ions.

- Covalent bonds form between elements that have similar electron negativities; the electrons are localized and shared equally between the atoms, leading to spatially directed angular bonds.
- Metallic bonds occur between elements with low electron negativities, so that the electrons are only loosely attracted to the ionic nuclei. A metal is thought of as a set of positively charged ions embedded in a sea of electrons.
- van der Waals bonds are due to the different internal electronic polarities between adjacent atoms or molecules, leading to weak (secondary) electrostatic dipole bonding forces.
- Material's spatial atomic structure: the amorphous or crystalline arrangement of atoms (or molecules) resulting from *long-range* or *short-range* bonding forces. In crystalline structures, it is characterized by unit cells which are the fundamental building blocks or modules, repeated many times in space within a crystal.
- Grains: crystallites made up of identical unit cells repeated in space, separated by grain boundaries.
- Phases: homogeneous aggregations of matter with respect to chemical composition and uniform crystal structure; grains composed of the same unit cells are the same phase.
- Lattice defects: deviations from ideal crystal structure.
 - Point defects or missing atoms: vacancies, interstitial or substituted atoms
 - Line defects or rows of missing atoms: dislocations
 - Area defects: grain boundaries, phase boundaries, and twins
 - Volume defects: cavities, precipitates.
- Microstructure: The microscopic collection of grains, phases, and lattice defects.

In addition to bulk materials characteristics, surface and interface phenomena also have to be considered.

In Fig. 1.9 an overview of the microstructural features of metallic materials is depicted schematically. Methods and techniques for the characterization of nanoscopic architecture and microstructure are presented in Chap. 5.

1.3.2 Types of Materials

It has been estimated that there are between 40 000 and 80 000 materials which are used or can be used in today's technology [1.14]. Figure 1.10 lists the main conventional *families* of materials together with examples of *classes*, *members*, and *attributes*. For the examples of attributes, necessary characterization methods are listed.

Metallic Materials and Alloys

In metals, the grains are the buildings blocks and are held together by the *electron gas*. The free valence electrons of the electron gas account for the high electrical and thermal conductivity, as well as for the optical gloss of metals. Metallic bonding, seen as the interaction between the total atomic nuclei and the electron gas, is not significantly influenced by displacement of atoms, which is the reason for the good ductility and formability of metals. Metals and metallic alloys are the most important group of the so-called *structural materials* whose special features for engineering applications are their mechanical properties, e.g., strength and toughness.

Semiconductors

Semiconductors have an intermediate position between metals and inorganic nonmetallic materials. Their most important representatives are the elements silicon and germanium, possessing covalent bonding and diamond structure; they are also similar in structure to III–V compounds such as gallium arsenide (GaAs). Being electric nonconductors at absolute zero temperature, semiconductors can be made conductive through thermal energy input or atomic doping, which leads to the creation of free electrons contributing to electrical conductivity. Semiconductors are important *functional materials* for electronic components and applications.

Inorganic Nonmetallic Materials or Ceramics

Atoms of these materials are held together by covalent and ionic bonding. As covalent and ionic bonding energies are much higher than those of metallic bonds, inorganic nonmetallic materials, such as ceramics, have high hardness and high melting temperatures. These materials are basically brittle and not ductile: In contrast to the metallic bond model, displacement of atomic dimensions theoretically breaks localized covalent bonds or transforms anion–cation attractions into anion–anion or cation–cation repulsions. Because of the lack of free valence electrons, inorganic nonmetallic materials are poor conductors of electricity and heat; this qualifies them as good insulators in engineering applications.

Organic Materials or Polymers and Blends

Organic materials, whose technologically most important representatives are the polymers, consist of macro-

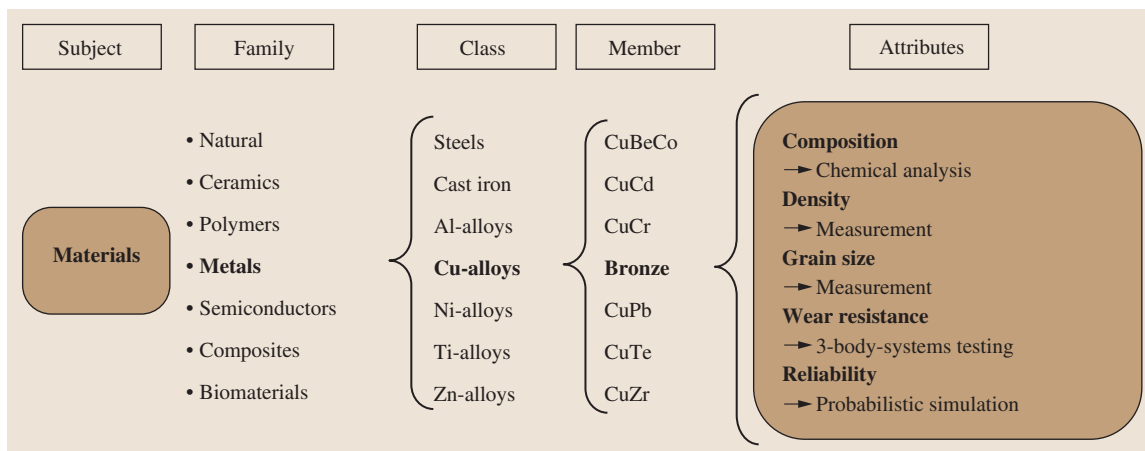


Fig. 1.10 Hierarchy of materials, and examples of attributes and necessary characterization methods

molecules containing carbon (C) covalently bonded with itself and with elements of low atomic number (e.g., H, N, O, S). Intimate mechanical mixtures of several polymers are called blends. In thermoplastic materials, the molecular chains have long linear structures and are held together by (weak) intermolecular (van der Waals) bonds, leading to low melting temperatures. In thermosetting materials, the chains are connected in a network structure and therefore do not melt. Amorphous polymer structures (e.g., polystyrene) are transparent, whereas crystalline polymers are translucent to opaque. The low density of polymers gives them a good strength-to-weight ratio and makes them competitive with metals in structural engineering applications.

Composites

Generally speaking, composites are hybrid creations made of two or more materials that maintain their identities when combined. The materials are chosen so that the properties of one constituent enhance the deficient properties of the other. Usually, a given property of a composite lies between the values for each constituent, but not always. Sometimes, the property of a composite is clearly superior to those of either of the constituents. The potential for such a synergy is one reason for the interest in composites for high-performance applications. However, because manufacturing of composites involves many steps and is labor intensive, composites may be too expensive to compete with metals and polymers, even if their properties are superior. In high-technology applications of advanced composites, it should also be borne in mind that they are usually difficult to recycle.

Natural Materials

Natural materials used in engineering applications are classified into natural materials of mineral origin, e.g., marble, granite, sandstone, mica, sapphire, ruby, or diamond, and those of organic origin, e.g., timber, India rubber, or natural fibres such as cotton and wool. The properties of natural materials of mineral origin, for example, high hardness and good chemical durability, are determined by strong covalent and ionic bonds between their atomic or molecular constituents and stable crystal structures. Natural materials of organic origin often possess complex structures with directionally dependent properties. Advantageous aspects of natural materials are ease of recycling and sustainability.

Biomaterials

Biomaterials can be broadly defined as the class of materials suitable for biomedical applications. They may be synthetically derived from nonbiological or even inorganic materials, or they may originate in living tissues. Products that incorporate biomaterials are extremely varied and include artificial organs; biochemical sensors; disposable materials and commodities; drug-delivery systems; dental, plastic surgery, ear, and ophthalmological devices; orthopedic replacements; wound management aids; and packaging materials for biomedical and hygienic uses. When applying biomaterials, understanding of the interactions between synthetic substrates and biological tissues is of crucial importance to meet clinical requirements.

1.3.3 Scale of Materials

The geometric length scale of materials covers more than 12 orders of magnitude. The scale ranges from

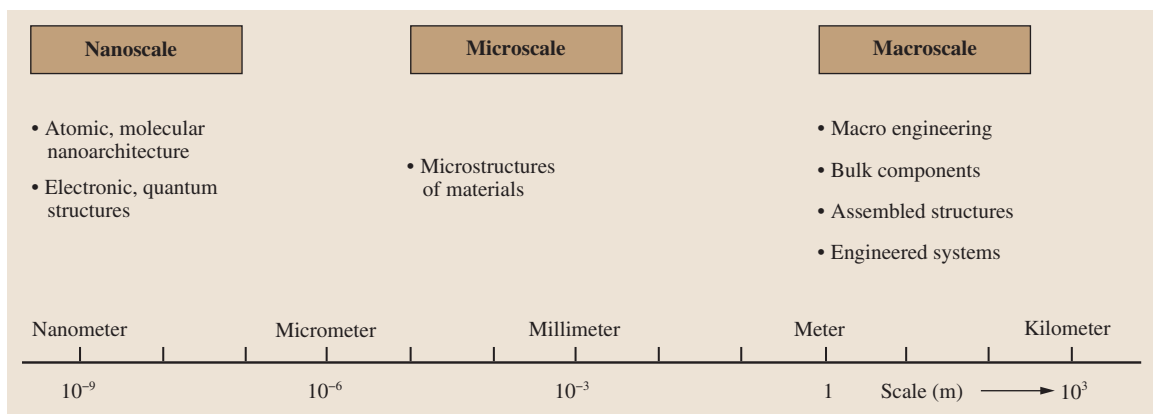


Fig. 1.11 Scale of material dimensions to be recognized in materials metrology and testing

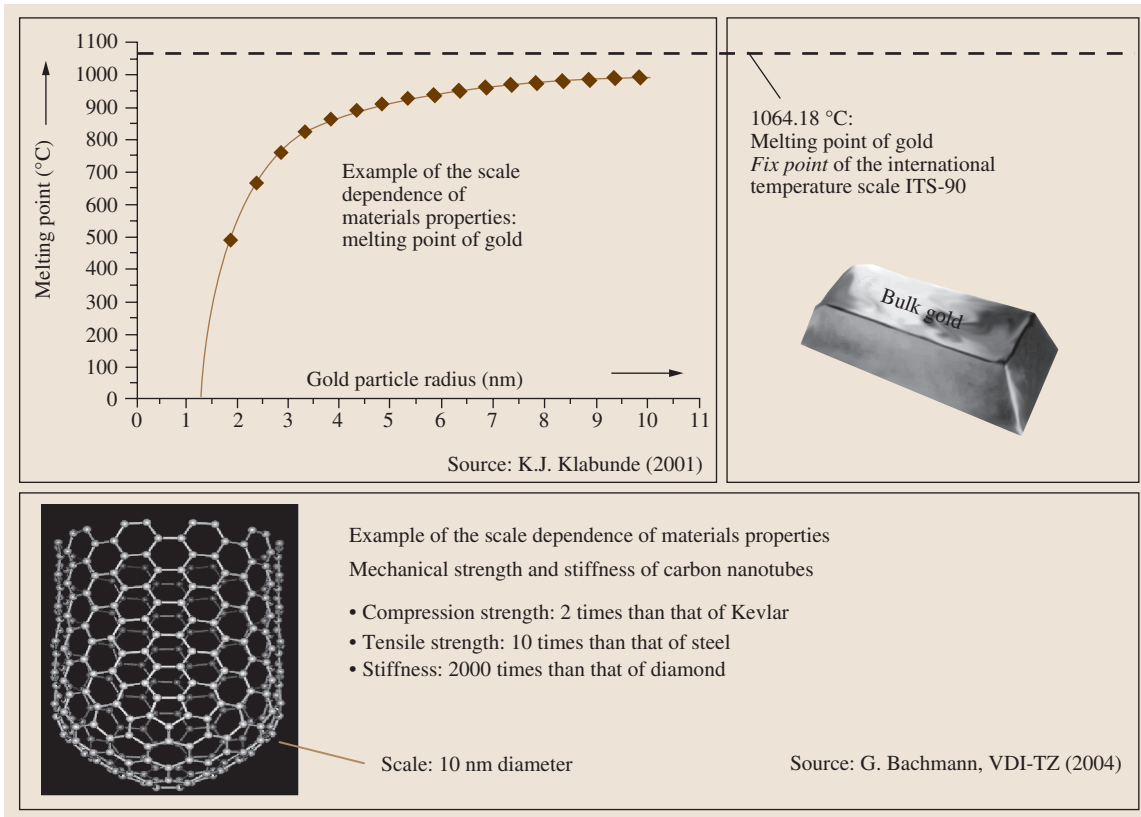


Fig. 1.12 Examples of the influence of scale effects on thermal and mechanical materials properties

the nanoscopic materials architecture to kilometer-long structures of bridges for public transport, pipelines, and oil drilling platforms for supplying energy to society. Figure 1.11 illustrates the dimensional scales relevant for today's materials science and technology.

Material specimens of different geometric dimensions have different bulk-to-surface ratios and may also have different bulk and surface microstructures. This can significantly influence the properties of materials, as exemplified in Fig. 1.12 for thermal and mechanical properties. Thus, scale effects have to be meticulously considered in materials metrology and testing.

1.3.4 Properties of Materials

Materials and their characteristics result from the processing of matter. Their properties are the response to extrinsic loading in their application. For every application, materials have to be engineered by processing, manufacturing, machining, forming or nanotechnology assembly to create structural, func-

tional or smart materials for the various engineering tasks (Fig. 1.13).

The properties of materials, which are of fundamental importance for their engineering applications, can be categorized into three basic groups.

1. Structural materials have specific mechanical or thermal properties for mechanical or thermal tasks in engineering structures.
2. Functional materials have specific electromagnetic or optical properties for electrical, magnetic or optical tasks in engineering functions.
3. Smart materials are engineered materials with intrinsic or embedded sensor and actuator functions, which are able to accommodate materials in response to external loading, with the aim of optimizing material behavior according to given requirements for materials performance.

Numerical values for the various materials properties can vary over several orders of magnitude for the different material types. An overview of the broad

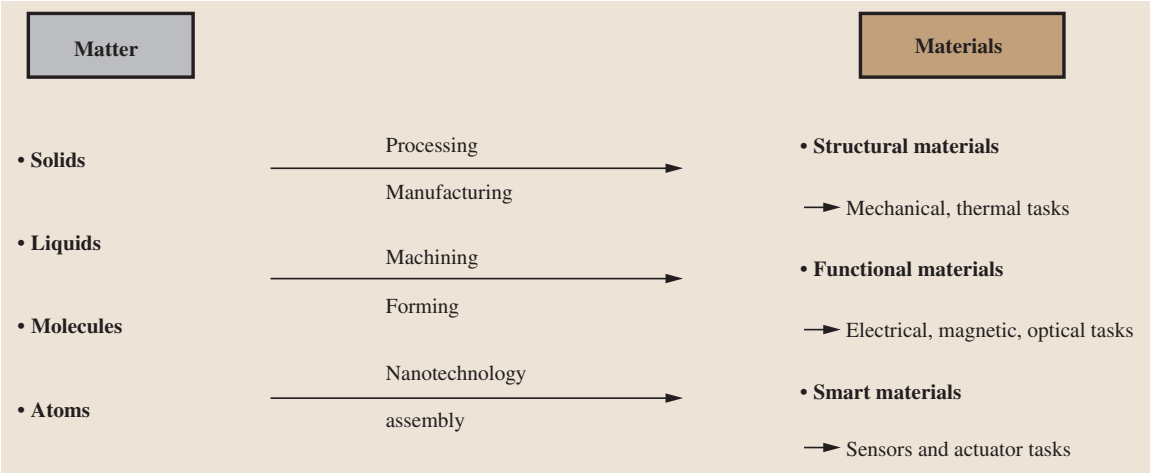


Fig. 1.13 Materials and their characteristics result from the processing of matter

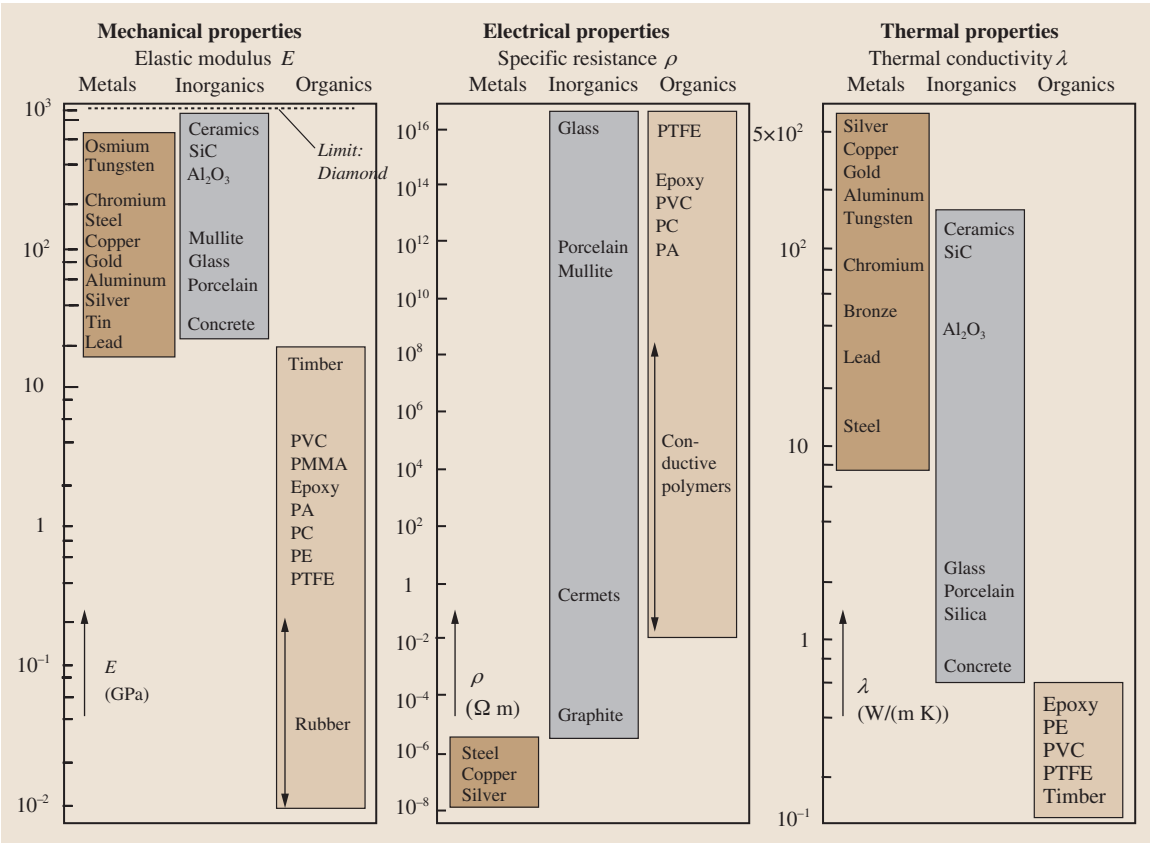


Fig. 1.14 Overview of mechanical, electrical, and thermal materials properties for the basic types of materials (metal, inorganic, or organic)

numerical spectra of some mechanical, electrical, and thermal properties of metals, inorganics, and organics is shown in Fig. 1.14 [1.16].

It must be emphasized that the numerical ranking of materials in Fig. 1.14 is based on *rough, average values* only. Precise data of materials properties require the specification of various influencing factors described above and symbolically expressed as

$$\begin{aligned} &\text{Materials properties data} \\ &= f(\text{composition–microstructure–scale,} \\ &\quad \text{external loading, } \dots) . \end{aligned}$$

1.3.5 Performance of Materials

For the application of materials as constituents of engineered products, performance characteristics such as quality, reliability, and safety are of special importance. This adds performance control and material failure analysis to the tasks of application-oriented materials measurement, testing, and assessment. Because all materials interact with their environment, materials–environment interactions and detrimental influences on

the integrity of materials must also be considered. An overview of the manifold aspects to be recognized in the characterization of materials performance is provided in Fig. 1.15.

The so-called materials cycle depicted schematically in Fig. 1.15 applies to all manmade technical products in all branches of technology and economy. The materials cycle illustrates that materials (accompanied by the necessary flow of energy and information) move in *cycles* through the technoeconomic system: from raw materials to engineering materials and technical products, and finally, after the termination of their task and performance, to deposition or recycling.

The operating conditions and influencing factors for the performance of a material in a given application stem from its structural tasks and functional loads, as shown in the right part of Fig. 1.15. In each application, materials have to fulfil technical functions as constituents of engineered products or parts of technical systems. They have to bear mechanical stresses and are in contact with other solid bodies, aggressive gases, liquids or biological species. In their functional tasks, materials always interact with their environment,

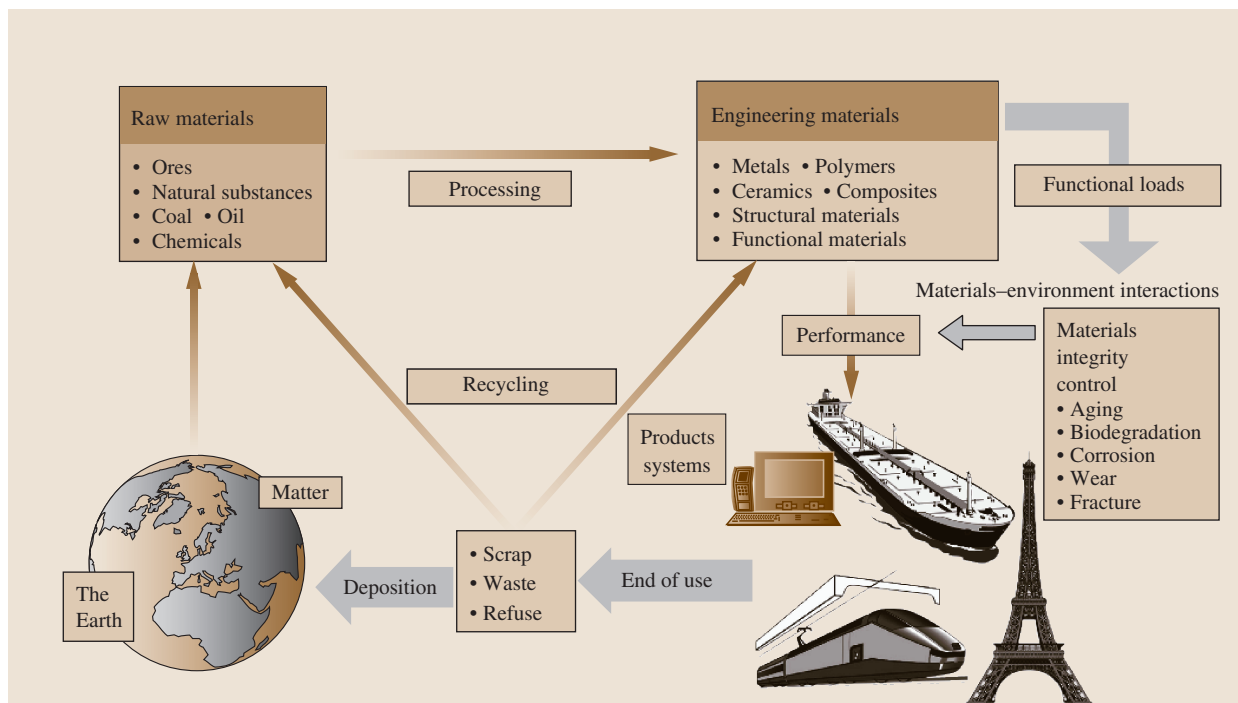


Fig. 1.15 The materials cycle of all products and technical systems

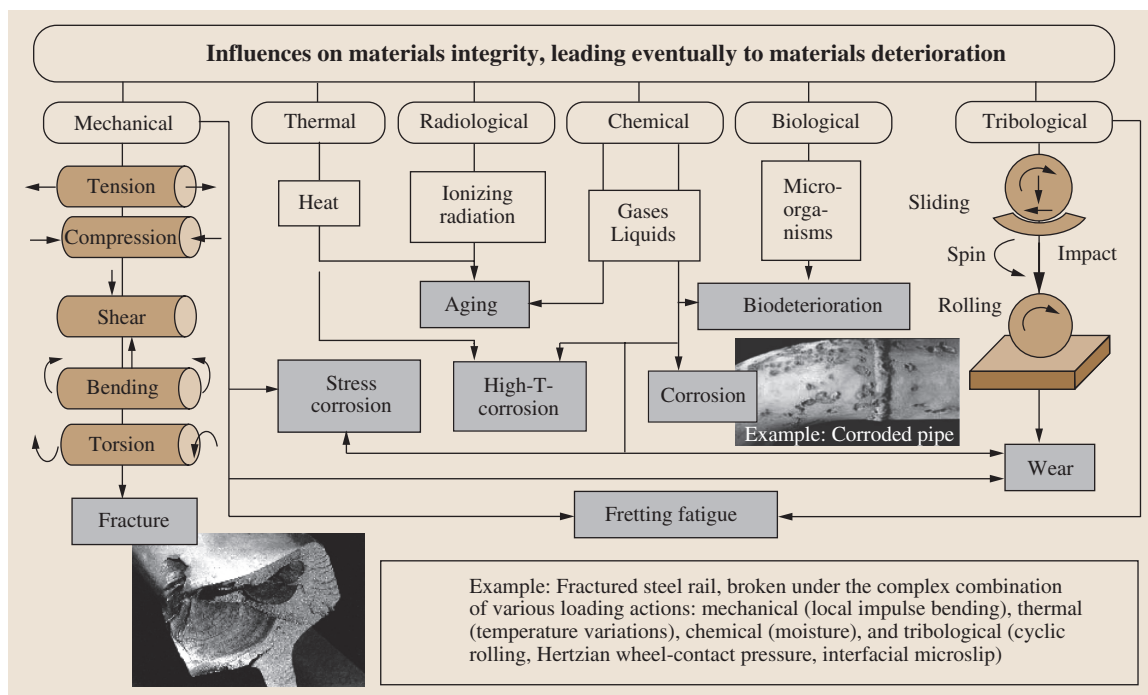


Fig. 1.16 Fundamentals of materials performance characterization: influencing phenomena

so these aspects also have to be recognized to characterize materials performance.

For the proper performance of engineered materials, materials deterioration processes and potential failures, such as materials aging, biodegradation, corrosion, wear, and fracture, must be controlled. Figure 1.16 shows an overview of influences on materials integrity and possible failure modes.

Figure 1.16 illustrates in a generalized, simplified manner that the influences on the integrity of materials, which are essential for their performance, can be categorized in mechanical, thermal, radiological, chemical, biological, and tribological terms. The basic materials deterioration mechanisms, as listed in Fig. 1.15, are aging, biodegradation, corrosion, wear, and fracture.

The deterioration and failure modes illustrated in Fig. 1.16 are of different relevance for the two elementary classes of materials, namely organic materials and inorganic materials (Fig. 1.8). Whereas aging and biodegradation are main deterioration mechanisms for organic materials such as polymers, the various types of corrosion are prevailing failure modes of metallic materials. Wear and fracture are relevant as materials deterioration and failure mechanisms for all types of materials.

1.3.6 Metrology of Materials

The topics of measurement and testing applied to materials (in short *metrology of materials*) concern the accurate and fit-for-purpose determination of the behavior of a material throughout its lifecycle.

Recognizing the need for a sound technical basis for drafting codes of practice and specifications for advanced materials, the governments of countries of the Economic Summit (G7) and the European Commission signed a Memorandum of Understanding in 1982 to establish the Versailles Project on Advanced Materials and Standards (VAMAS, <http://www.vamas.org/>). This project supports international trade by enabling scientific collaboration as a precursor to the drafting of standards. Following a suggestion of VAMAS, the Comité International des Poids et Mesures (CIPM, Fig. 1.6) established an ad hoc Working Group on the Metrology Applicable to the Measurement of Material Properties. The findings and conclusions of the Working Group on Materials Metrology were published in a special issue of *Metrologia* [1.17]. One important finding is the *confidence ring for traceability in materials metrology* (Fig. 1.4).

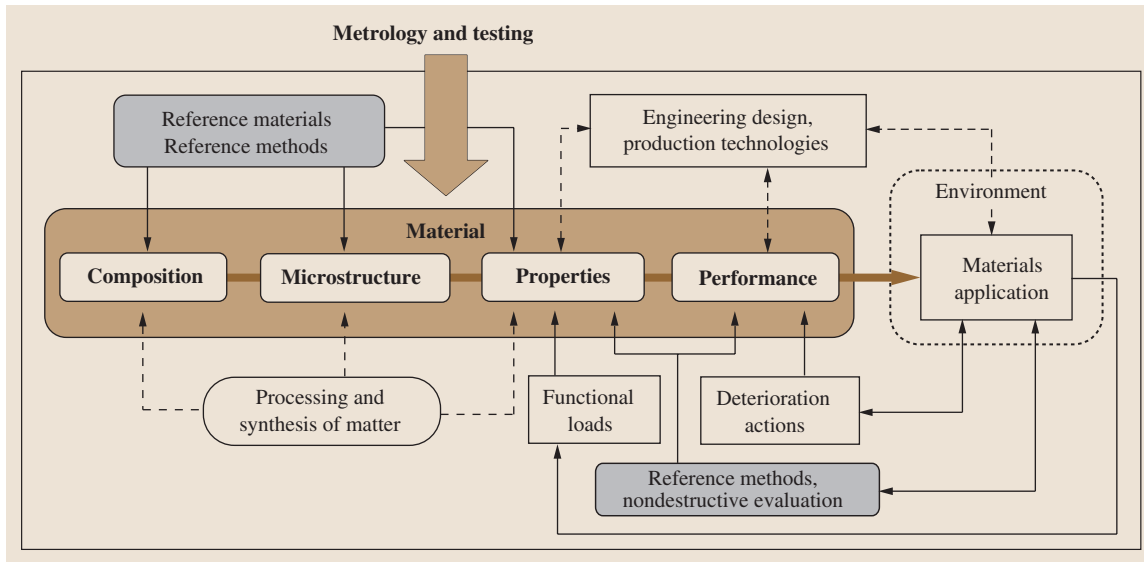


Fig. 1.17 Characteristics of materials to be recognized in metrology and testing

Materials in engineering design have to meet one or more structural, functional (e.g., electrical, optical, magnetic) or decorative purposes. This encompasses materials such as metals, ceramics, and polymers, resulting from the processing and synthesis of matter, based on chemistry, solid-state physics, and surface physics. Whenever a material is being created, developed or produced, the properties or phenomena that the material exhibits are of central concern. Experience shows that the properties and performance associated with a material are intimately related to its composition and structure at all scale levels, and influenced also by the engineering component design and production technologies. The final material, as a constituent of an engineered component, must perform a given task and must do so in an economical and societally acceptable manner. All these aspects are compiled in Fig. 1.17 [1.15].

The basic groups of materials characteristics essentially relevant for materials metrology and testing, as shown in the central part of Fig. 1.17, can be categorized as follows.

- *Intrinsic characteristics* are the material's composition and material's microstructure, described in Sect. 1.3.1. The intrinsic (inherent) materials characteristics result from the processing and syn-

thesis of matter. Metrology and testing to determine these characteristics have to be backed up by suitable reference materials and reference methods, if available.

- *Extrinsic characteristics* are the material's properties and material's performance, outlined in Sects. 1.3.4 and 1.3.5. They are *procedural characteristics* and describe the response of materials and engineered components to functional loads and environmental deterioration of the material's integrity. Metrology and testing to determine these characteristics have to be backed up by suitable reference methods and nondestructive evaluation (NDE).

It follows that, in engineering applications of materials, methods and techniques are needed to characterize intrinsic and extrinsic material attributes, and to consider also structure–property relations. The methods and techniques to characterize composition and microstructure are treated in part B of the handbook. The methods and techniques to characterize properties and performance are treated in parts C and D. The final part E of the handbook presents important modeling and simulation methods that underline measurement procedures that rely on mathematical models to interpret complex experiments or to estimate properties that cannot be measured directly.

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