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The critical role of materials metrology in engineering

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[Plate 1]

This paper analyses the critical underpinning role of materials research and testing for contemporary engineering and future technologies. It is obvious that measurements, tests and evaluations of materials and components, i.e. materials metrologies, are crucial to provide the information and data needed to optimize the function of engineering structures. The industrial needs and research trends in materials metrology are reviewed, and recent BAM-developments of measuring techniques for high-technology sectors, like high-temperature technology or high-performance ceramics, are presented. Finally, the relevance of an appropriate metrological base for the establishment of industrial standards, agreed codes of practice and the harmonization of test procedures for the international trade of technical products is discussed.

1. Introduction

The key role of materials technologies for future industrial developments has been recognized in all industrialized countries in recent years (Hondros 1985). This can best be illustrated by reviewing the major materials developments identified already in the 1979 U.S. National Academy of Sciences five-year outlook as being those likely to have a major impact on society until the turn of the century (National Academy of Sciences 1979). The list includes the following materials and materials-related developments: synthetic polymers; high-performance resin- and metal-matrix composites; silicon nitride and silicon carbide ceramics; rapidly solidified superalloys; single-crystal engine components (e.g. turbine blades); fibre-optic transmission; very-large-scale, integrated-circuit silicon chips; bubble memories for computers; semiconductor infrared detectors; powder metallurgy consolidation techniques; precision casting; laser treatment of surfaces; computer-aided design and manufacturing.

The driving forces behind the materials developments are various technological, social, and environmental requirements, for example: improved performance, integrity and reliability of engineering systems; higher durability of products; higher-efficiency, lower-energy consuming engines; light-weight, high-strength structures; high-speed information technologies; increased productivity of manufacturing technologies.

To fulfil these requirements, there is a continuous need to develop materials with specific functions: mechanical, thermal, optical, electromagnetic, chemical, biological and electronic. In addition, accurate and reliable measuring and testing techniques are needed to provide the data and information required to characterize the materials, their properties and the engineering functional behaviour. It follows, that as an important basis for contemporary engineering, the simultaneous availability and application of (i) materials, (ii) properties and (iii) metrology is needed as illustrated in a highly simplified manner in figure 1.

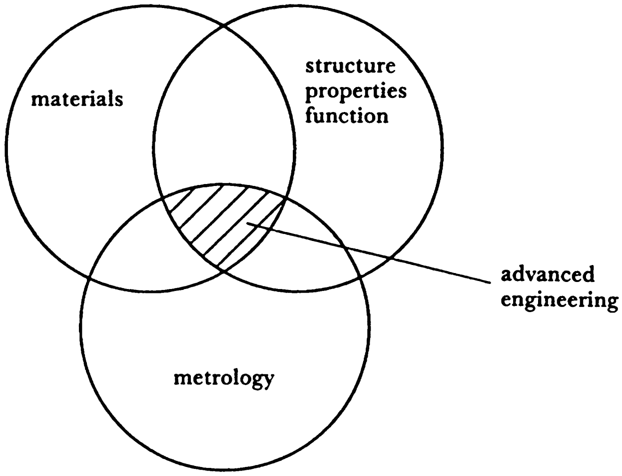

FIGURE 1. Materials-related requirements for advanced engineering.

2. RESEARCH AND DEVELOPMENT TRENDS

Research and development trends in (i) materials, (ii) relevant material properties and (iii) metrological tools for future technological developments can be deduced from the results of a pertinent study within the European Communities (Czichos 1982).

In 1982 on behalf of the Commission of the European Communities (EEC), a study was carried out to determine the future materials research requirements within the EEC. Questionnaires were sent to trade associations, scientific and technological societies in all main branches of industry and materials technology, in which respondents were asked to indicate the areas of materials research and testing and the technically or industrially important material properties. In addition to this survey, a campaign of consultation was conducted in the Community Countries taking the form of individual discussions and interviews with key persons having good overall knowledge of the present situation and future requirements in materials research and testing in the Community Countries concerned. Research suggestions of about 200 bodies in industry and among the universities and research establishments were summarized in the BAM Research Report (Czichos 1982) in terms of the following technological aspects: (i) materials category; (ii) materials properties; (iii) measuring and testing techniques; i.e. materials metrology. Tables 1–3 show the distribution of research suggestions in these areas (multiple nominations were allowed). The results of tables 1–3, which are self-explaining,

TABLE 1. RESEARCH SUGGESTIONS ON MATERIAL CATEGORIES WITHIN THE EUROPEAN COMMUNITIES

material category	proportion of research suggestions (%)
composite materials	58
metals and alloys	51
ceramics, inorganic materials	46
organic materials	32
energy and information-relevant materials	14
renewable materials	7
biomedical materials	5

TABLE 2. RESEARCH SUGGESTIONS ON MATERIAL PROPERTIES WITHIN THE EUROPEAN COMMUNITIES

material properties	proportion of research suggestions (%)
corrosion behaviour	40
wear and tribological properties	28
thermal behaviour	28
strength	26
durability (chemical, biological, environmental)	22
fatigue	17
fracture mechanics	15
long-term stability, creep	11
surface properties	7

TABLE 3. RESEARCH SUGGESTIONS ON MEASURING AND TESTING TECHNIQUES WITHIN THE EUROPEAN COMMUNITIES

measurement and testing techniques	proportion of research suggestions (%)
corrosion studies	33
tribological tests	26
non-destructive testing	25
fracture mechanics and crack investigations	19
strength tests	18
surface investigations	12
thermal stability studies	12
computer aided materials testing	11
fatigue investigations	11
quality control	7
determination of service life	6
long-term stability and creep	6
adhesive strength testing	6
structural analysis	6

illustrate real industrial needs with respect to material categories and properties and underline the central role of materials metrologies: from table 3 it can be seen that the research suggestions clearly emphasize important metrological areas and cover a very broad spectrum, ranging from corrosion studies to structural analyses. Although it is not possible within the scope of this paper to deal with details of the various metrological developments compiled in table 3, several overall research and development trends in the field may be summarized as follows (National Science Foundation 1984; Hirschfeld 1985).

(i) Expansion of measurement and test techniques to the frontier of extreme physical or environmental conditions: for example, temperature, pressure, materials purity.

(ii) Experimental and theoretical approaches for the careful study of model systems which isolate certain fundamental or technological important problems.

(iii) Increasing sophistication and power of theoretical tools to confront the complexity of real materials.

(iv) Experimental developments for the fuller characterization of materials on the atomic scale, which ultimately determines the macroscopic properties of primary interest.

(v) The need for an improved understanding of the relations between engineering performance of materials (or materials deterioration) and the composition and microstructure.

(vi) An increased focus on experimental and metrological tools for the better understanding and controlling the processes used to make materials, and the tailoring of specific properties either for practical applications or fundamental study.

(vii) Improvements of measurement and test equipment, marked by a considerable level of 'intelligence' through built-in or connected computers, microminiaturization, and sensor-based instrumentation allowing practice-oriented online investigations.

3. Advanced materials metrology: examples of BAM developments

To illustrate the central enabling role of measurements in contemporary engineering, specific examples of advanced materials metrology developments will be presented for two important 'high-tech' sectors, namely high-temperature technologies, and advanced materials, such as ceramics.

3.1. *High-temperature metrology of position and strain*

High-temperature technologies are of great interest for future technological developments in connection with attempts to increase the efficiency of engineering systems, such as turbines or power plants. A crucial metrological problem concerns the long-term accurate measurement of position and strain of mechanical components at temperatures exceeding 800 °C. At such temperatures, conventional position and strain gauges show serious disadvantages because of the temperature-dependence of relevant material properties that lead to intolerable drifts. At BAM, a fluidic measurement system with a good long-term stability has been developed (Mayer 1984). The fluidic system is operated with an inert gas for the signal generation and transmission. It consists of four nozzles arranged in a circuit analogous to the well-known Wheatstone bridge, see figure 2. As illustrated in figure 3, the measuring quantity, *s*, moves

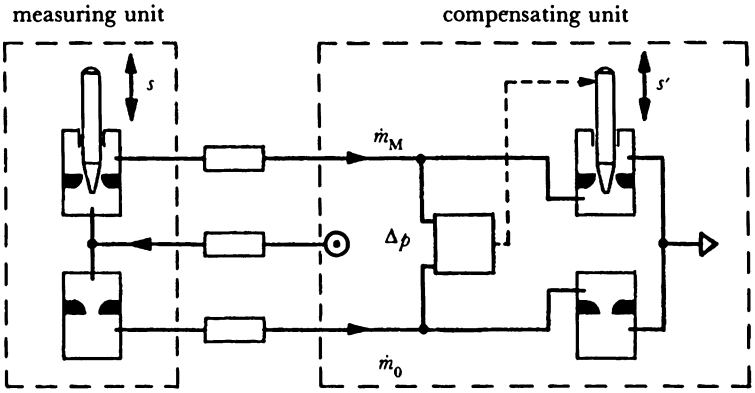

Figure 2. Block diagram of fluid-measurement system for position and strain at high temperatures. $\dot{m}_0$, Reference mass flow; m_M variable mass flow; Δp, gas pressure.

a needle, d, which controls the mass flow of the gas through one sensor nozzle, b. A corresponding needle is used to balance the bridge automatically. Because there is critical flow through the sensor nozzles, the system is very insensitive to pressure and temperature fluctuations. The system operates at position ranges of 0.2–2.5 mm, with a resolution of better than 1 μm at the 0.2 mm range. Figure 4 shows that the long-term stability – as registered over about 450 days – is in the narrow range of ±1 %.

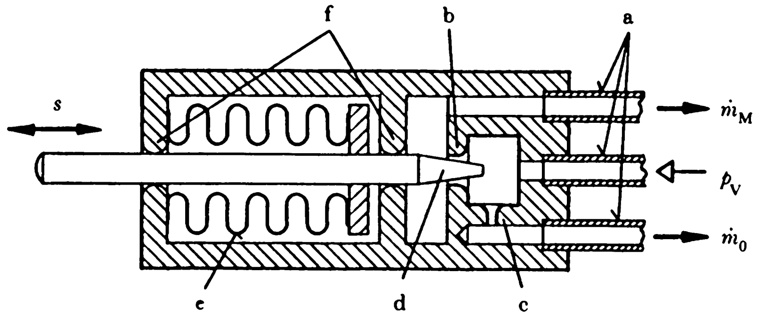

FIGURE 3. Design of the transducer. a, Metal tubes; b, sensor nozzle; c, reference nozzle; d, needle; e, metal bellow; f, guides; $\dot{m}_0$, reference mass flow; $\dot{m}_M$ variable mass flow.

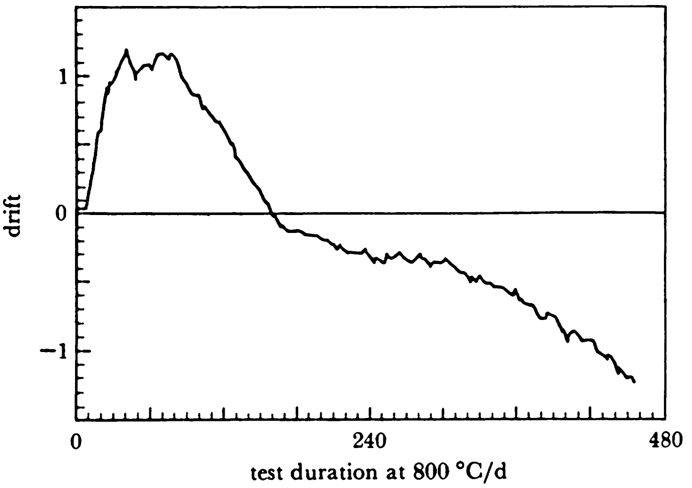

FIGURE 4. Long-term stability of fluidic measurement system for position and strain at high temperatures.

3.2. *Computer-aided non-destructive techniques* (NDT) *for advanced materials*

Ceramic materials are of great interest for future technologies because they promise great thermal stability, low mass and beneficial corrosion and wear behaviour. However, ceramic materials may contain flaws, like cracks, pores, and inclusions, which may be detrimental to the functional behaviour.

At BAM, computer-aided non-destructive testing and evaluation techniques for the detection of flaws in advanced materials, for example ceramics, have been developed on the basis of (i) X-ray computerized tomography and (ii) ultrasonic testing, UT.

The BAM computer tomograph consists of an X-ray radiation source (Co 60) of 420 kV, a high-precision three-dimensional specimen stage (maximum object diameter 1 m; maximum mass 1000 kg) and a detector system of 31 photomultipliers with plastic scintillators. The computer tomograph produces through the successive X-ray scanning of an object under different angles, maps of the local X-ray absorption inside an object, i.e. a three-dimensional picture of the interior of the object. Figure 5 shows the block diagram of the CT scanner, data acquisition, and processing (Goebbels *et al.* 1986).

In figure 6 the NDT inspection of flaws in an SSiC-rotor (sintered silicon carbide) is shown. In (*a*) a projection image (not a tomogram) of the rotor is shown; the white lines in the lower part indicate the location of the performed cross-sectional tomograms of the rotor. In (*b*) a

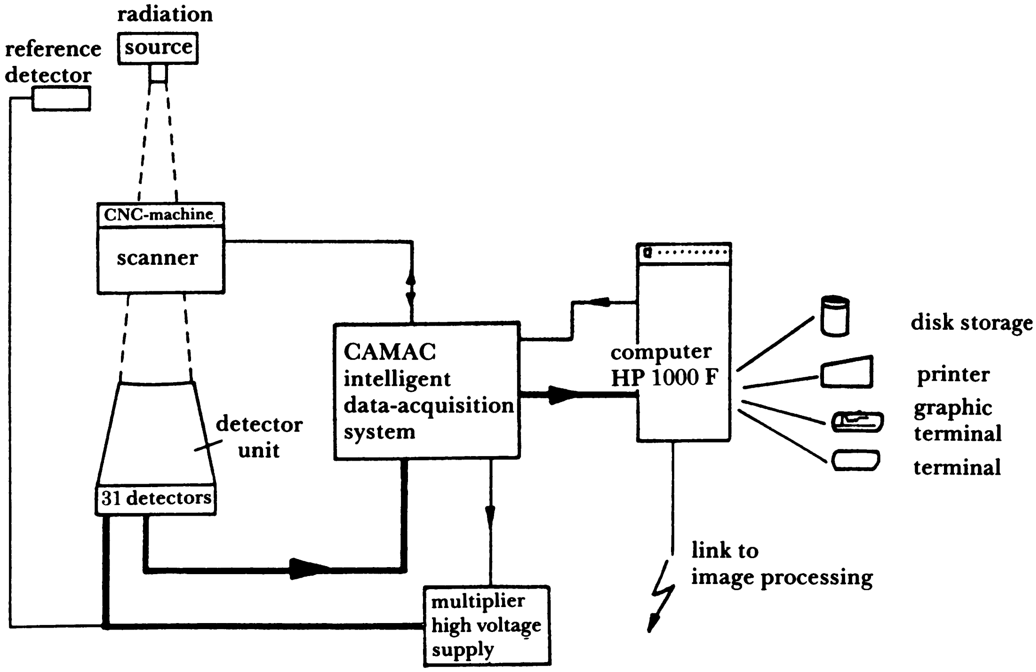

FIGURE 5. Block diagram of computer tomograph, data acquisition and processing.

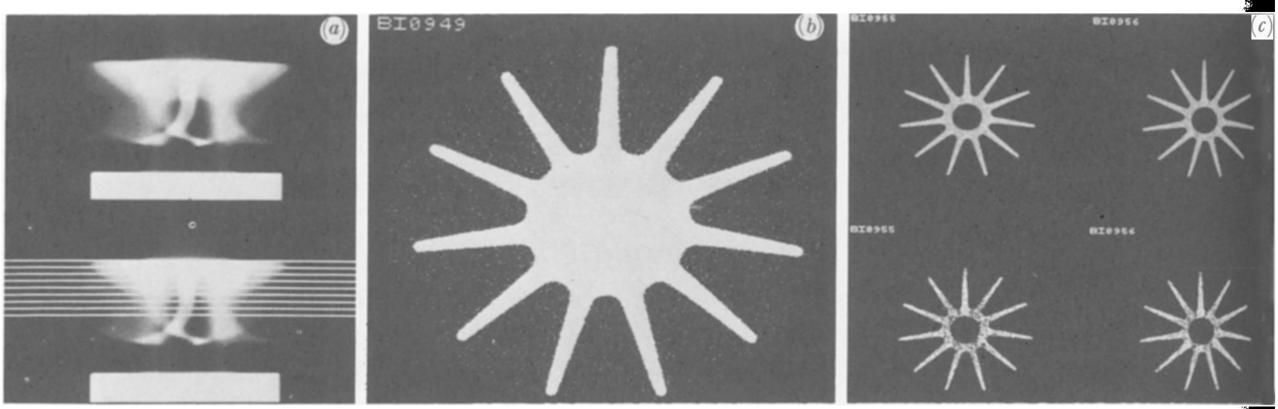

FIGURE 6. Inspection of a SSiC-rotor by computer tomography. (*a*) Projection image of the rotor, (*b*) tomogram of the rotor (location of fourth white line from the top of (*a*), (*c*) tomogram of the rotor (location of two lowest white lines of (*a*).

tomogram of the rotor at the location of the fourth white line from the top of (*a*) at a grey level range of 0–20 (from 255 grey levels) is shown. In (*c*) two tomograms at locations of the two lowest white lines of (*a*) are shown. The lower part of these two tomograms shows one, respectively two, voids in the wheel nave. Because of the very good density resolution of the BAM computer tomograph, defects in ceramics can be detected and located with a width of about $\frac{1}{10}$ of the spatial resolution, which is in the range of about 200 μm.

In addition to the computer tomograph based on radiography, an advanced multipurpose ultrasonic test (UT) equipment has been developed that can be applied to various types of

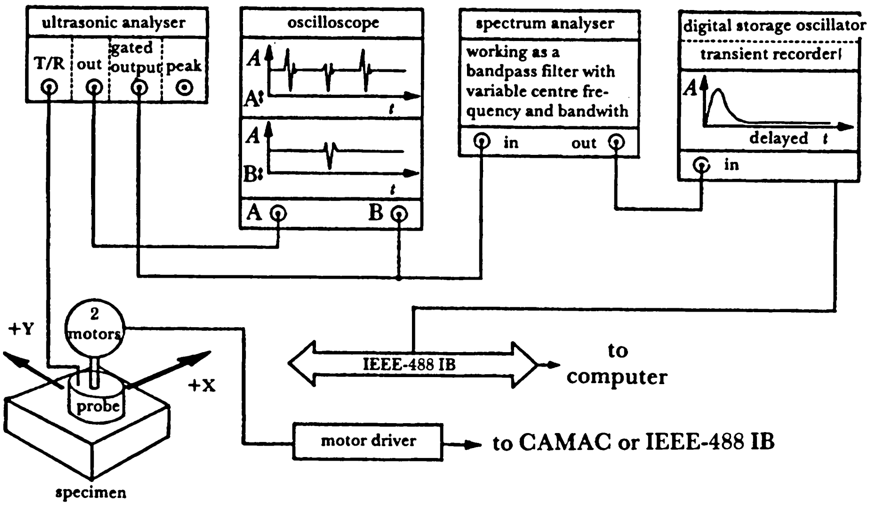

FIGURE 7. Block diagram of ultrasonic test equipment.

materials (Hecht *et al.* 1986). The components of the UT equipment, see figure 7, are commercially available and may be combined in variable arrangements to realize different UT-techniques. By using this measuring equipment, ceramic materials can be inspected non-destructively in a way that cannot otherwise be achieved. Figure 8, plate 1, illustrates the detection of a Si-vein in an SiSiC (silicon-infiltrated silicon carbide) plate, 10 mm thick, by UT with a focusing transducer of 25 MHz. The lower part of figure 8 shows a photograph of the cross section of the SiSiC plate. By using ultrasonic testing, a defect indication at different probe positions is possible. Through destructive follow-ups it could be shown that with the NDT technique the different types of flaws can be detected as compiled in figure 9, plate 1. Of special importance was the NDT detection of the crack-like Si-vein (figure 9*c*) because the specimen cracked exactly along the location of the vein under mechanical bending stresses.

4. MATERIALS METROLOGY AND STANDARDIZATION

To apply materials metrology to engineering, standardization of techniques and methodologies is essential. Thus, all industrialized countries have clearly recognized the prime importance of standards and test methods as a means of encouraging market penetration of new materials and of products containing new materials. The broad and important scope of materials-related standardization is obvious from a brief review of the standardization system in Japan, the U.S.A. and the F.R.G.

In Japan, by law the deliberation of Japanese Industrial Standards (JIS) is the duty of the Japanese Industrial Standards Committee (JISC), which is attached to the Agency of Industrial Science and Technology (AIST) (Agency of Industrial Science and Technology 1985). Promotion of industrial standardization has contributed industrial infrastructures and has improved Japanese industries' rationalization of production together with the implementation of standards through quality control measures in factories. Japanese industrial standards are established by the cooperation of some 8000 experts from industry and academic circles as well as consumers. Because the Japanese industrial standards are voluntary standards they essentially reflect the opinions of all concerned.

The Japanese industrial standards can be classified into the following three categories.

(i) Product standards: shape, dimensions, quality, performance, etc. (about 5000).

(ii) Methods standards: testing, analysis, inspection and measuring (about 2000).

(iii) Basic standards: terminology, symbols, units, etc. (about 1000).

At the end of 1984, the number of Japanese industrial standards reached 7931, see tables 4 and 5.

TABLE 4. MATERIAL STANDARDS IN JAPAN (AIST 1985)

	number	percentage
ferrous materials and metallurgy	329	19.7
non-ferrous metals and metallurgy	346	20.8
pulp and paper	102	6.2
ceramics	227	13.6
fundamentals and general	660	39.7
total	**1664**	**100.0**

TABLE 5. INDUSTRIAL STANDARDS IN JAPAN (AIST 1985)

	number	percentage
chemical engineering	1587	25.3
mechanical engineering	1167	18.6
electrical engineering	819	13.2
shipbuilding	524	8.4
civil engineering and architecture	516	8.2
automotive engineering	340	5.4
textile engineering	303	4.8
domestic wares	260	4.2
mining	235	3.7
railway engineering	219	3.5
medical equipment and safety appliances	209	3.3
aircraft and aviation	88	1.4
total	**6267**	**100.0**

In the U.S.A., the American Society for Testing and Materials, ASTM, is the leading standardization organization in the field of materials and materials-related technologies (Gerischer 1985). It was founded in 1902 and has now some 29000 members. Within the U.S.A. there are about 50 organizations that develop industry standards; the ASTM is one of the largest of those organisations.

At the beginning of 1985, the ASTM had issued 7340 standards, which are classified in table 6.

The ASTM tries to update its standards at regular intervals; all committees are requested to critically review their standards every five years to reconfirm them or to delete them. This requirement is obviously followed by the various committees because, for example, 94% of the standards of class E in table 6 are not more than six years old.

In the F.R.G., the Deutsches Institut für Normung (DIN) is the national standardization organization. It covers all areas of technologically or industrially relevant areas of

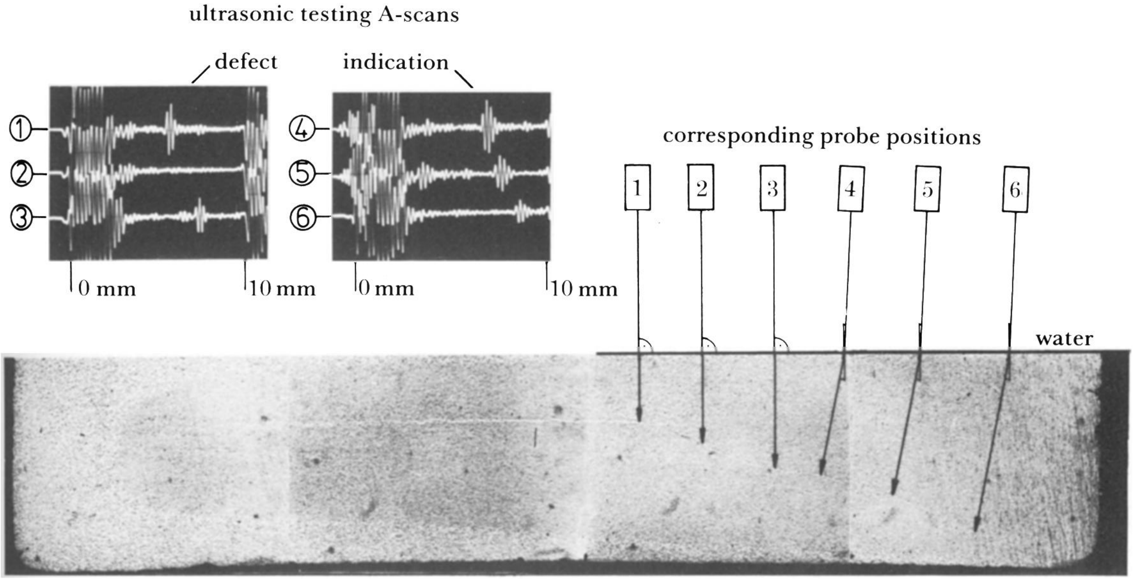

FIGURE 8. Detection of a Si-vein in SiSiC by UT with 25 MHz focusing transducer.

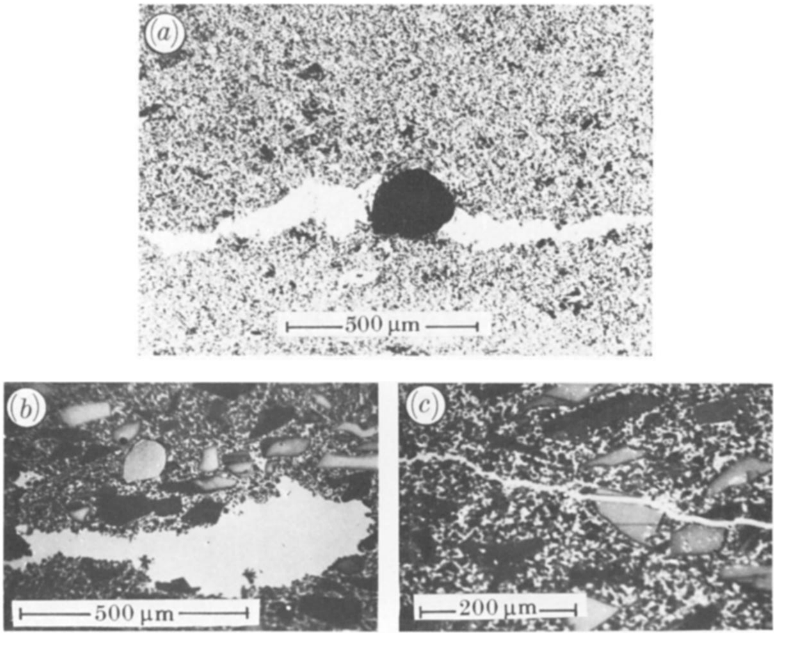

FIGURE 9. Different types of flaws in SiSiC detectable by UT with 25 MHz focusing transducer. (*a*) Single pore and Si-enrichment, (*b*) local Si-enrichment, (*c*) crack-like Si-vein.

(*Facing p.* **368**)

standardization. Within the DIN, the Normenausschuss Materialprüfung (NMP) is responsible for the standardization in the field of materials testing (Rauls 1983). Similarly, as the ASTM, the DIN committees combine people from industry, research institutes and universities to work out standards for the various fields. At present, the NMP within the DIN has more than 200 individual committees working on the development of standards in the various fields of materials and materials testing. In table 7 a classification of the existing standards within the DIN system of testing and evaluating of materials is given.

TABLE 6. MATERIAL STANDARDS IN THE U.S.A. (ASTM 1985)

	number	percentage
ferrous metals	616	8.4
non-ferrous metals	635	8.6
cementitious ceramics, concrete and masonry materials	918	12.5
miscellaneous materials	3524	48.0
miscellaneous subjects	835	11.4
materials for specific applications	716	9.8
corrosion, deterioration and degradation of materials	96	1.3
total	**7340**	**100.0**

TABLE 7. STANDARDS ON MATERIALS TESTING IN THE F.R.G. (DIN 1986)

	number	percentage
metals	150	8.3
non-metallic inorganic materials	306	16.9
refractories, ceramic materials	103	5.7
organic materials	342	18.9
elastomers	160	8.8
pulp and paper	92	5.1
timber and wood-based materials	131	7.2
corrosion and wear	74	4.1
non-destructive testing	39	2.2
miscellaneous	413	22.8
total	**1810**	**100.0**

5. MATERIALS METROLOGY AND PRESTANDARDIZATION RESEARCH: THE VAMAS PROJECT

In the past few years the production of goods worldwide has been characterized by a high degree of innovation. Many, perhaps most, of these recent advances in technology depend on the development of novel materials. Thus new materials characterization and performance measurements as well as new testing concepts and procedures are needed concurrently with the appearance of these materials in commercial technology. However, the achievement of the traditional long basis of experience before the need for standards arises is not possible for advanced materials. Thus, the rapid growth in the manufacture and use of highly engineered materials, together with the promise of even greater advances in the future have placed severe strains on existing materials-performance standards, both national and international. Interest in advances of contemporary technologies and concern with their implications has been particularly intense in the industrialized countries represented at the Economic Summits in which Canada, France, Germany, Italy, Japan, the U.K., the U.S.A. and the Commission of the European Communities participate.

As a result, in 1982 the Heads of State meeting at the Economic Summit in Versailles considered the various technical sources of expansion in the world economy. The leaders identified the following critical generic areas as subject for international collaboration to improve technology, growth and employment:

(1) advanced robotics;
(2) aquaculture;
(3) biotechnology;
(4) controlled thermonuclear fusion;
(5) fast breeder reactors;
(6) food technology;
(7) high-speed trains;
(8) photosynthesis and photochemical conversion of solar energy;
(9) photovoltaic solar energy;
(10) remote sensing from space;
(11) *advanced materials and standards*;
(12) basic biology;
(13) high-energy physics;
(14) solar-system exploration;
(15) housing and urban planning in developing countries;
(16) impact of new technology on mature industries;
(17) new technologies applied to culture, education and vocational training;
(18) public acceptance of new technologies.

For every area, steering committees have been formed of representatives of each of the participants in the Economic Summit.

For the Versailles Project on Advanced Materials and Standards, VAMAS, leadership by the U.K. and the U.S.A. was designated to alternate between the two countries every three years (Schwartz & Steiner 1987). The VAMAS project is a scheme to stimulate the introduction of advanced materials into high-technology products and engineering structures with the overall aim of encouraging international trade therein (VAMAS 1985) through (*a*) multilaterial research aimed at furnishing the enabling scientific and metrological base necessary to achieve agreement on standards; (*b*) international agreement on codes of practice and performance standards.

Within the project on advanced materials and standards, the activities identified in the following along with the leading country have now been initiated:

wear-test methods (Germany);
surface-chemical analysis (U.S.A.);
ceramics (France);
polymer blends (Canada);
bioengineering materials (Italy);
hot-salt-corrosion resistance (U.K.);
polymer composites (France);
superconducting and cryogenic structural materials (Japan);
weld characteristics (U.S.A.);
factual materials databanks (U.S.A. and EEC);
high-temperature mechanical properties (U.K.).

Activity in these areas includes

(i) establishment of priorities for prestandards activity;
(ii) consultation and collaboration in prestandards research;
(iii) intercomparison of specific measurements in a number of governmental and commercial laboratories.

6. Materials metrology and the international trade

For many years government purchasing authorities and large industrial consumers of goods, materials, and services have operated on the various forms of national standards and quality requirements aimed at providing assurance that the goods or services supplied meet the purchaser's specifications. However, in recent years it has been increasingly recognized that the various national standardization systems if not internationally harmonized form serious obstacles to the free international trade of technical products (Schneider 1984). Several countries have now introduced or are now developing accreditation schemes that seek to provide uniform standards by which the competence of laboratories to perform tests on materials and technical products may be judged. The term 'laboratory accreditation' is taken to mean a formal recognition that a testing laboratory is competent to carry out specific tests or specific types of tests. Such an accreditation is granted only after the accrediting organization is satisfied that the particular laboratory meets all specific criteria. Accreditation procedures currently adopted provide for

(i) initial assessment of aspects of laboratory management and operation by panels of expert ascessors;

(ii) reassessment at prescribed intervals;

(iii) proficiency testing or other forms of objective audit programmes (where possible) on a regular basis.

Laboratory accreditation systems take various forms because they have been designed to meet particular national and local means. In 1977 an annual International Laboratory Accreditation Conference, ILAC, has been started in connection with the General Agreement on Tariffs and Trade, GATT, in cooperation with the European Community, EGC, the Organization for Economic Cooperation and Development, OECD, and the International Standardization Organization, ISO. ILAC is seeking to harmonize the criteria for accreditation adopted by systems in each participating country and to reach international agreement on the terms used in laboratory accreditation. In the long term it is the aim of ILAC to achieve international recognition of test results with the basic objectives of enhancing the validity of tests, promoting confidence in test data, facilitating trade and commerce, and making more efficient use of testing facilities and resources.

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