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C O N T E N T

Investigation of materials' properties

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1. INTRODUCTION

Modern quality control is always concentrated on the investigation of the materials properties which are related to the field of application of the material. For special fields it is therefore necessary to develop new methods or to modify existing methods respectively. In this report it is not possible to report all the recently developed or modified methods, because of the great number of tests dus to the increasing number of more specific application.

In order to give an idea about interesting and specific trends and tendencies two groups of investigation and tests are reported in detail, both characterize typical developments in this field.

The properties may divided in

- mechan'cal
- electrical
- optical

and thermophysical properties

The various trends in general should be explained by the description of the first, the mechanical, and the last, the thermophysical group of properties.

The methods for the determination of the mechanical properties are well established even for advanced material and a great number of international standard tests exist already. Great efforts are made to adapt these tests for specific fields too and for a fully automatical procedure.

The chapter about the mechanical tests therefore includes the general trends preferably and gives some examples of

interesting specific methods, which can applied for various types of materials.

The second chapter which deals with thermophysical properties gives a general description of the development of the methods used, which are still further improved. One of the main tests in this field is the improvement of the experimental equipment to achieve a higher accuracy, that means more reliable data. Errors of $\pm$ 5 % are relative low because often the data of one material reported in the literature are scattered within one order of magnitude.

In former time simple experimental arrangements were sufficient, but recently the engineers and designer need more accurate data and therefore more sophisticated equipment is necessary. Especially with regard to the problems of energy saving slight improvements in isolation have to be measured because they can result a remarkable economic effect.

There are only few methods which have become an international standard already but the number of standard tests is increasing.

In this report the field of developments of measuring methods shall be described in an extended way, to illustrate the trends in this modern field of quality control. Nevertheless, these methods are also intended to run fully automatically.

2. MECHANICAL PROPERTIES

Mechanical properties are still one of the most important ones in quality control, because resistance against mechanical impact mostly determine the lifetime.

According this importance tests to measure the tensile strength, the hardness, the ductility and so on were developed long time ago and are now routinely used. In spite of new methods they

will be used in the future too.

Mechanical tests play also an important role in the quality control of advanced materials. Due to the specialities of these materials, their structure, specific properties and fields of application, it is often necessary to adapt or modify the existing methods to the special requirements. There are a great number of such modifications of course if you concern the different types of advanced materials such as fibre reinforced composites, rather brittle ceramics, glass fibre reinforced plastics, high temperature resistant and high ductile metallic alloys and so on. Therefore in this paper some typical methods, which probably are of more general interest, are described.

Independent of the material to be examined recent developments in the field of mechanical testing are concentrated on two tasks.

One is to test the materials under conditions, which are very close to the practice that means on the one hand to experimentally determine the properties under conditions such as high temperatures or corrosive athmospheres and on the other hand to test not only small specimens of material but structure elements such as tubes or even more complicated designed elements. Reason of this recent trend is the increasing safety consideration as well as economical considerations. The more reliable the data the more accurate are the calculations of design and structure and therefore the smaller the safety factor.

Second which is in some way also connected to safety considerations is the intension to a fully automatic control of the measurement proceoure. Beside an economical reason which is probably not valid for developing countries, the reason for automatic control is the much more better reproducibility of the test procedure. Therefore as it is used in other fields of science and technology in quality control computers and data acquisition systems become more and more important.

According the application of microcomputers for data acquisition and evaluation in materials testing the methods and apparatus are changed. Nowadays an automatical performance of the measurement procedure, the data acquisition and evaluation can be afforded even by small laboratories. In addition the application of microelectronics enables the design of compacted portable apparatus, which can be used outside the laboratories too. Time consuming procedures such as calibration of measurement systems, compensation of disturbing influences can be carried out by the suitable software.

There are four main tasks for the electronics.

First and most important is the data acquisition. An automatical data acqusition system is most valuable or even necessary, if one have to deal with a very large number of measuring points and/or a large number of data. Second is the evaluation of the data, which includes the reduction of data, calculations using predetermined formulae and the documentation of process parameters and of the results. For the first two tasks personal computers are mainly used.

Third an automatic control which is necessary to make the test performances highly reproducible. The controlling parameters may be the time, certain test parameters, the temperature, the stress and something else or given limits. Fourth is the control of the process parameters, that means the stabilization of the temperature the control of a temperature programm, load cycles, gas composition and so on.

In addition there is another positive aspect using data acquisition systems in materials testing. Connected with a central computer the data can be transfered to this unit stored there and recalled by other technical or administrative departments. For instance the production can get a quick response of the product's quality or certificates can be plotted.

TENSILE TEST

This test is the well known standard test for the measurement of mechanical properties.

By the means of the modern electronic equipment this test has not been changed in principle but the evaluation of the stress-strain diagramm is now much more easier to evaluate and therefore it is possible to get more information out of the results of this simple test procedure. Computers control the test procedure, calculate the characteristic values such as tensile strengths, fracture thoughness and so on, store the data, compare them with other results and perform statistic calculations. Statistics are very important to get informations about the reliability of the average values and the scattering band.

NOTCHED BAR IMPACT TEST

The impact test measures the fracture thoughness in a very quick way. Unfortunately often the results obtained in the impact test are not able to characterize the bulk material quantitatively. Therefore the use of the impact test is mainly reduced to the investigation of sheet materials, in which field it is also a standard test.

To get more reliable information from this test a modern trend of this well established method is the instrumentation of the test. Instrumentation arises a new problem , it is that disturbing vibrations with high frequencies are superimposed on the transient load-time respectively load-deflection record. The problem was investigated and it could be shown that the predominante vibration modes are the longitudinal vibrations of the hammer blade between the tup and the backside of blade. Because of these results a modified hammerblade has been designed with special precaution

for increased damping of the disturbing vibrations. It could be experimentally shown that most of the disturbing vibrations are removed. This will improve the evaluations of the transient load-deformation-diagrams of an instrumented notched bar impact test because the characteristic points of the diagrams can be identified in a better way.

HARDNESS TEST

The well known tests are the Rockwell, Vickers and Brinell test. They are very commonly used because they can characterize the mechanical properties in a very simple and quick way. Therefore there will be no remarkable change of these tests in the future. Nevertheless the apparatus are improved and specialized for modern requirements. Microprocessors and computers are applied to hardness tests too. There are already fully autometic tester available. A further improvement is the reduction of weight of the apparatus that they are portable. Such devices are specially suitable for the performance of rapid and reliable hardness tests i.e. on site testing of large and heavy work pieces or fixed installation components, during production, especially during mass production, for machines already in place, at material storage depots for the identification of materials, at difficulty accessible locations and in cramped space conditions and for testing variations in hardness of especially large workpieces.
In addition to the experimental improvements great efforts are made to get a maximum of information from the simple hardness test. Therefore formula are developped and calculations are made to get a most chacteristic value of the material from the geometric dimensions of the impression in the test piece..
The investigation of microhardness become more popular. Because of the measuring demand of these investigations developments are made to run this test automatically controlled too. The intensions were successful, that there is an automatic apparatus commercially available already.

CREEP AND FATIGUE

Continuous deformation can occur in materials and in particular in metals and alloys at high temperature, when they are subjected to constant applied force. This time-dependent deformation is called creep. The criteries for alloy development for high temperature application e.g. gas turbines are mainly the creep properties. When the creep characteristics of a material are well established, an engineering component can usually be designed to achieve the required life under specified operating conditions without failure due to creep.

The most widely used technique for providing engineering data involves the tensile creep rupture test machine. This consists of a dead weight applied to a lever system acting on a tensile test-piece of cylindrical or sheet shape with the gauge portion heated by a resistance furnace. Temperature, load, rupture time and rupture elongation are the parameters measured.

If strain versus time relationships have to be measured a strain recording system is needed. For this purpose test-pieces with ledges at the two ends of the gauge length are usually adapted. The relative displacement of the ledges is transfered out of the furnace and then measured. High sensitivity inductive or capacitor transducers have replaced mechanical and optical systems, thereby facilitating automatic data logging and their computer elaboration. If fast strain variations or a highly disturbed strain signal have to be measured a continuous recording, usually by a chart recorder, is better than logging pairs of discrete values of strain and time.

Fatigue data currently obtained and used in engineering design and performance assessment are endurance data, usually in the form of an S-N (S.....stress N.....number of cycles) or ε-N

(ε.....strain) curve. Endurance curves are derived from constant amplitude tests on plain specimens or structural features.

In recent time hydraulic closed-loop and computer-controlled machines for mechanical testing have become available. This modern, versatile and precise equipment permits complex tensile, compression and bending stress cycles to be performed and is widely employed for fatigue but also for creep at variable load, for creep fatigue and for stress relaxation tests.

A further aspect directed to life time prediction is thermal fatigue. Most low-cycle fatigue problems in high temperature machinery involve thermal as well as mechanical loadings. By thermal loadings it is meant that the material is subjected to cylic temperature simultaneous with cylic stress. Analysis of these loadings and consideration of the attendant fatigue becomes very complex. Recent advances in analysis methods, e.g. finite element computer programs and testing equipment e.g. servohydraulic test system help to solve the problems.

ACOUSTIC EMISSION

Acoustic emission is called a quasi non destructive testing because these may be effects in the specimen which lead to a damage of the material. Nevertheless it becomes more often used in material testing. In general acoustic emission can be defined as: The elastic wave generated by the release of energy internally stored in a structure. Acoustic emission "sources" which can be described as different processes emitting elastic waves, can be basically classified in 4 different groups

- dislocation movements
- phase transformations
- friction mechanismus
- crack formation and extensions

Related to mechanical properties the crack formation and extension is the most important one.
The highest amplitudes of the emitted signal are also generated from crack formation.
It occurs at surface notches or at points inside a material where local stresses exceed the fracture stress. The crack formation results in creation of new surfaces and strain energy is released which is partly transformed to acoustic emission signals. If the source emits a spherical wave packet, it will be propagated as such only in an infinite isotropic, homogeneous, ideally elastic medium. In real structures the propagation will be effected by surfaces, grain boundaries, microcracks, inclusions etc. anisotropy, inhomogenities and non-linear elastic behaviour.
When the emitted stress wave reaches the transducer the stress-strain condition has to be converted into an electrical signal which can be treated by electronic means. After amplification and a proper evaluation of the signal an even detailed knowledge of the nature of the source can be obtained. A detailed analysis of the waveform, the frequency spectrum and the amplitude distribution is often necessary.
Some of the advantages of the method are remote detection and location of flaws, integral method, high sensitivity and detects active flaws. On the other hand some of the limitations are noted, high dependence on materials and coupling method, difficulties to detect spourious sources, limited information on the type of flaw.

Because of the possibilities of the method it is already used in practice for testing different kind of advanced materials too.

A seperate, very important field of quality control is the testing by nondestructive methods (NDT), but because of its great extent it is not possible to cover it in this report too. The great number of tests, which are still increasing, may be divided according the physical properties the tests are based on.

- optical methods (holographical methods)
- radiological methods (absorption, diffraction, transmission, reflexion of particles and radiation respectively)
- nuclear physical methods (activation analysis)
- elastic oscillation (acoustic waves, ultrasonic tests)
- magnetical and electrical methods (scattering and diffraction of microwaves or magnetic field, eddy current tests)

For details the related literature has to be studied.

3. THERMOPHYSICAL PROPERTIES

DENSITY AND POROSITY

Density and porosity are important parameter to characterize the condition of a material. Often the knowledge of theoretical density and the porosity is sufficient for a rough estimation of the properties of porous materials. A great number of properties are influenced by the porosity e.g. thermal conductivity, electrical conductivity, some mechanical properties etc. Especially with regard to the advanced materials such as high-tech ceramics and powder metallurgy (P/M) materials the values of porosity play an important role and indicate the quality of the products.

At the beginning, the definition of density and porosity should be noted

- bulk density: The mass of the material divided by its bulk volume, i.e. its total volume including internal pores
- true density: The mass of the material divided by the volume of solid material excluding open porosity.
- theoretical density: The density of the material excluding all forms of porosity.
- total porosity: The volume of void space within the material divided by the bulk volume, expressed as a fraction or percentage.
- open porosity: The volume of void space within a material accessible from the exterior, divided by the bulk volume.
- closed porosity: The volume of void space within the material inaccessible from the exterior, divided by its bulk volume.

The bulk density of samples of simple geometric forms such as cylinders or cubes can be easily determined from their dimensions and weight.

To determine the true density the displacement principle is used. Modern pycnometry represents a refinement of the displacement principle and often uses a gas as the displaced medium. In the gas pycnometry the volume displacement is determined from the pressure-volume relationship of a gas under controlled conditions. Helium is recommended instead of a liquid because it does not adsorb on most materials, can penetrate pores as small as 0,1 mm and behaves as an ideal gas. Further there are no problems with wetting, interfacial tension and evaporation.

Another method for density measurement is based on the absorption of a mono-energetic X-ray or γ-radiation. The rate of a absorption depends beside the thickness from wave length, atomic number of the sample material and density. Wave length atomic number and

thickness can kept constant. The resulting constant (K) can be determined by calibration with a material of well-known density. The density of the specimen material can be calculated by the following expression

$$\rho = \frac{\ln (I/Io)}{-K}$$

I ..... intensity of the radiation
Io ..... initial intensity of the radiation

The theoretical density of cystalline materials is possible to calculate from the characteristics of the elementary cell, that means their number of atoms (Z) and the structure typ, the distance between atomic planes (a) and the atomic weight (A). For a cubic crystal the theoretical density (d) is given by the expression

$$d = \frac{A.Z}{N.a^3}$$

where A/N is the weight of one single atom (N..... Loschmitt number). The required structure data are obtained by X-ray structure analysis.
A more specific method is the levitation method, which is used to measure the density of powders and small particles. The material is dispersed in a liquid of which the density is continuously changed by the addition of a second liquid of different density. Levitation of the particles indicates that their density is equal the density of the liquid mixture. The latter one can easy be measured by the determination of volume and weight.

Total porosity is difficult to measure. One approach is to grind the material to a powder finer than the smallest inter-space distance and comparing the powder density with the bulk density. Measuring the density of the generated powder one has to take care that the fine powder particles do not trap bubbles during immersion in the liquid of the pycnometer. Another way to determine the total porosity is to compare the bulk density

with the theoretical value measured on the same material or taken from the literature. Alternatively, the total porosity can be estimated by a line intercept or area counting method on carefully prepared polished cross-section.

A suitable and recently common used method to measure the <u>open porosity</u> is the mercury porosimetry. This method uses the penetration of porous solids by mercury under pressure to characterize the pore volume and pore size distribution of interconnected pores ranging from 500 to 0.003 μm in diameter. The porous material to be analysed is placed into a mercury porosimeter, in which the volume of mercury absorbed by the pores is measured as a function of the pressure applied to the mercury. The technique has been applied to a variety of porous materials, such as concrete, bricks and tiles, engineering ceramics, porous metals and powders. The method is based on the following equation:

$$d = \frac{-4\gamma \cos\vartheta}{p}$$

Where P is the pressure required to force the mercury through a pore of diameter d. ϑ is the contact angle between the mercury and the porous material. γ is the surface tension of mercury. Mercury is used as the intrusion liquid because it does not wet most materials, a fundamental requirement of the method.

THERMAL CONDUCTIVITY AND DIFFUSIVITY

Heat flow and temperature distribution determine the energy consumption. According to the importance of energy saving the thermo-physical properties become more and more important and a great effort is made to measure the thermal conductivity of insulating as well as of good conductive materials in an accurate way.

In the experimental determination of the thermal conductivity of solids, a number of different methods of measurement are required for different ranges of temperatures and for various classes of materials having different ranges of thermal conductivity values. A particular method may thus be preferably over the others for a given material and temperature range, and no one method is suitable for all the required conditions of measurement. The appropriateness of a method is further determined by such considerations as the physical nature of the material, the geometry of samples available, the required accuracy of results and the speed of operation.

Two measurement principles are the basis for all the methods for conductivity determination. First the method with steady state heat flow, in which the test specimen is subjected to a temperature profile which is time invariant. The thermal conductivity is determined directly by measuring the rate of heat flow per unit area and by measuring the temperature gradient after equilibrium has been reached.
Second the non-steady state method, in which the temperature in the specimen varies with time. Using this method, the rate of temperature change is measured. From these data and the geometric dimensions the thermal diffusivity can be determined. The thermal conductivity K is then calculated from the thermal diffusivity a, the density d, and the specific heat Cp of the material according the formula

$$K = a.d.Cp$$

Some of the methods commonly used in order to determine the conductivity of advanced materials are described in more detail.

Guarded hot plate.
This steady state method is preferably used for low-conductive materials such as fibre insulating material, high porous samples

and compacted powders.

Shape and size of the specimen used in various apparatus are different, but in general the specimen length to width ratio is small, because the smaller this ratio the smaller is the ratio of lateral heat losses to the heat flow to the specimen. The absolute value of the diameter of a disk shaped specimen commonly used may even exceed 1 m.

The experimental arrangement is so designed that the flow of heat is only in one direction, i.e. the axial direction of the cylindrical specimen. The radial heat losses are prevented or minimized in most of apparatus by the use of a guard heater, which is so adjusted that the temperature gradient is zero in all directions except in the direction of the axial heat flow.

Provided that the above mentioned requirement are fullfilled the thermal conductivity can be determined by the following equation

$$K = \frac{-q \Delta x}{A \Delta T}$$

K ..... thermal conductivity

q ..... rate of heat flow

A ..... cross sactional area of the specimen

ΔT..... temperature difference between two points of measurement along the heat flow directions

Δx..... distance between the points of temperature measurement

In most of the modern apparatus the rate of heat flow is determined by the powerinput to an electrical heater, but it may be also determined by a calorimeter or a heat flow meter. The temperature measurements are made generally with thermocouples inserted in the specimen or embedded in grooves on the specimen surfaces. The method can be fairly good used up to about 400^{0} C.

The guarded hot plate method is very well developed and very often used in research institute as well as in the laboratories of the industry. According its range of applicability it is the most important method for the determination of every kind of insulating material for technical application and buildings.

Recent work to improve the method was mainly directed to evaluate the heat losses, which are not completely negligible, in order to correct the obtained results and increase their accuracy.

Investigations are also carried out running the apparatus under different atmospheres, because the kind of gas and its pressure influence the conductivity of porous insulating material remarkably.

Comparative method

This method is also based on a longitudinal steady state heat flow. The rate of heat flow is determined by the temperature drop in a reference material. The reference sample of known thermal conductivity is placed in series with the unknown specimen. Under ideal conditions, that means the same rate of heat flow through the reference sample and the specimen, the thermal conductivity can be determined by the use of the following formula

$$K = K_r \frac{A_r (\Delta T/\Delta x)_r}{A (\Delta T / \Delta x)}$$

where the subscript r designates the reference sample.

Comparative methods have the advantages of simpler apparatus and easier operation. Because of these benefits they are very suitable in the field of material development, since they can quickly provide thermal conductivity data of new materials, especially the increasing number of composite materials can easily be tested by this method.
The results obtained are of lower accuracy than results by the hot plate method due to the additional uncertainly in the conductivity of the reference sample, the conductivity mismatch between specimen and reference sample and due to the interfacial thermal contact resistance. The method is usually used in the temperature range of -50°C up to 950°C. Above 950°C it is very difficult to reduce the heat losses, that they are neglectable.

For special purposes methods with radial steady state heat flow were also developed. One cylindrical method is the most important one of these methods but is also used only for special applications, e.g. thermal conductivity measurements of granules or powders, because of the rather complicatad specimen preparation.
The conductivity is calculated from the expression

$$K = \frac{q \ln (r_2/r_1)}{2\pi l (T_1-T_2)}$$

l ....length of the centr-l heater
T_1, T_2 ....temperatures measured at radii r_1 and r_2

Line heat source method

This method uses a long thin heater wire as line source. The wire usually made of Platinum is supplied by a constant power input. This hot wire - therefore the method is often called "hot wire method" - is embedded in a large specimen, which is initially at

uniform temperature. After the heater is turned on the temperature of the hot wire is recorded as a function of time.

The thermal conductivity is given by the expression

$$K = \frac{q}{4\pi l} \cdot \frac{\ln (t_2/t_1)}{(T_2-T_1)}$$

T_2-T_1 .....temperature difference between two different times (t_2, t_1)

Main advantage of this method is the applicability up to very high temperatures. Therefore the method plays an important role in energy saving investigations because it is very suitable for the determination of thermal insulating materials for high temperature application such as refractory stones. The large specimen volume (dimensions are: 230 mm x 114 mm x 64 mm) makes it suitable for the determination of heterogenous materials and even of powder samples. The disadvantage of the method is the very long time for a test and the limited conductivity range ($K < 2$ W $m^{-1}K^{-1}$) - if you do not measure the temperature of the hot wire itself, but on a certain point in the sample, it is possible to increase the conductivity range up to 25 W $m^{-1}K^{-1}$).
The method is to become an international standard method for testing refractories. A german prestandardisation exists already (DIN 51 046) and a ISO standard is under elaboration.

Flash method

By the use of this method the thermal diffusivity is determined. In this method a flash of thermal energy is supplied to one of the surfaces of a disk specimen within a time interval that is short compared with the time required for the resulting transient flow of heat to propagate through the specimen. In most of the modern apparatus a laser supplies a flash of energy to the front face of a thin disk specimen. The dimensions of the specimen depend

of the design of apparatus used but are in general about 10 mm diameter and 1 to 3 mm height. At the rear face of the specimen the temperature time history is recorded. It is advantageous to measure the temperature by a contactless method such as by pyrometry in order to avoid the problems with thermal contacts, response time and heat transport along the wires using thermocouples. The thermal diffusivity can be determined from the thickness of the specimen l and the time $t_{1/2}$ at which the backface temperature reaches half its maximum value by the expression

$$a = 0.139\ l^2/t_{1/2}$$

Advantages of this method are the very simple specimen geometry, simple operation, very quick measurements and a wide temperature and diffusivity range. Because of these advantages it isavery suitable method to generate the data for a data sheet and it is also a very proper quality control instrument.

Flash diffusivity measurements are therefore often used in the field of material development work, e.g. very recently for the investigation of materials for cutting tools, cemented carbides as well as ceramics. Another field of application is connected to safety considerations related to nuclear power stations. In that case it is important to know the temperature distribution in a reactor core after a loss of coolant accident. In order to calculate this distribution the thermal diffusivity of Uranium dioxide at very high temperatures and even in the molten state have to be known.

Almost all of the materials can be easy measured by the laser flash method. Care has to be taken determining heterogeneous materials, because representative sampling and the achievement of an

unidirectional heat flow may be difficult. Translucent materials can be only measured by this method if the faces are overcoated with an opaque heat absorbing layer. The method is a standard for diffusivity determination of graphite samples up to high temperatures. In addition work is going on to establish it as an international standard (ASTM e 37.05.03) for diffusivity measurement in general. The thermal conductivity can be calculated from diffusivity, specific heat and density data as mentioned before.

THERMAL EXPANSION

Thermal expansion data are necessary for the calculation of thermal stresses. In addition this property very well characterize the material, e.g. the determination of phase transition and the shrinkage during the sintering process are important applications of thermal expansion measurements.

The trend in advanced materials is to develop materials with very low thermal expansion coefficient. Such materials are very suitable for high temperature application because of the reduced thermal stresses and thermal shock sensitivity. On the other hand the development of these materials requires measuring methods, which are able to determine even very low thermal expansion at very high temperature.

The linear expansion coefficient is the fractional increase in length per degree rise in temperature at a particular temperature and therefore given by the following expression

$$a\,(T) = \frac{1}{l}\,\frac{dl}{dT}$$

a.....linear expansion coefficient

l.....length

The mean expansion coefficient, which is commonly reported is given by the equation

$$a = \frac{1}{Lo} \quad \frac{L - Lo}{T - To}$$

i.e. fractional increase in length as a result of increase in temperature from To to T.

Tube dilatometers

These dilatometers are the most commonly used ones. With the non-differential tube type dilatometers the method is a comparison method in which the thermal expansion of the specimen is compared to the tube material. With a differential dilatometer the thermal expansion of a specimen is measured relative to a reference material.

An important application for differential expansion measurements is for quality control. It is used now routinely for quality control measurements for instance on gas turbine components and on glass-ceramics. Developments had to be made to run the dilatometers at high temperatures too. High temperature dilatometers are nowadays made of alumina, tantalum or other refractory materials.

Interferometers

With interferometric methods the absolute value of thermal expansion can directly be measured. The number of fringes that pass a reference point is a measure of the change in length of the specimen. Using these methods it is necessary to polish parallel optical flats on two opposing faces of a rigid test block and to take measurements of displacement after several hours of stabilization of temperature. The accuracy of the mean thermal expansion coefficient is limited by uncertainty in temperature uniformity but it is in the range of 10^{-8} K^{-1} over the temperature range of 25 up to 200°C. Because of the requirement on the sample condition porous or thin bars can not be measured by these techniques.

X-ray methods

The thermal expansion of crystalline materials can also be measured with X-ray cameras and diffractometers. The principle of the measurement is based on the change of the distance of atomic planes with temperature. With the means of X-ray diffraction the quantity of this change can be measured.
When using the camera method the specimen has to be a fine-grained polycrystalline wire, a powder or a single crystal.
Instead of X-ray neutron diffraction may used for special measurements of thermal expansion of materials, which are composed of light elements such as special plastics and other hydrocarbon based material. The main disadvantage compared with X-ray method is the price because for thermal neutrons one needs a nuclear reactor as source.

SPECIFIC HEAT

Specific heat also characterize the thermophysical-thermodynamical behaviour of the material and it is necessary for the calculation of thermal conductivity from diffusivity measurement and vice versa. Therefore a description of some important methods is included in this report.

The primary methods for the measurement of the specific heat of solids which are commonly used are the methods of mixtures or drop method, adiabatic method, comperative method, pulse heating method and modifications of these.

Drop calorimetry

In a drop calorimetry the heated sample is dropped in the calorimeter, where the heat given off by the sample to the environment is measured. According the medium to which the heat is released the calorimeter is called ice, isothermal water or copper block calorimeter. The amount of heat released from the specimen is measured by the temperature increase of the medium e.g.

water or copper.

Pulse heating method

For materials which are electrical conductors this method may be preferably used. The specimen is directly heated by its resistance. The current flowing through the specimen and the voltage drops are measured.simultaneously as a function of time. The specific resistance at each time interval is calculated from the cross-sectional area of the sample, the voltage, the current and the distance between voltage probes. The specific electrical resistance is then plotted as a function of time. The specific heat can be calculated from the mass of sample, the temperature coefficient of the resistance and the time rate of change of resistivity.

Differential Scanning Calorimeter

It is an apparatus commonly used to determine the specific heat often it is used to generate the data especially for conversion thermal diffusivity in thermal conductivity data and vice versa. Small specimens - solid samples are often cylindrical shaped with a diameter of about 6 mm and a height of about 1 mm - are fastened on a heated sample holder and the heat input are measured to achieve a linear increase of specimen temperature. The calibration is checked with a reference material usually a sapphir is used as standard.

4. RECOMMENDATIONS, SUMMARY

Because of the increasing economical relationship between industrilized and developing countries the number of goods, which the developing countries have to sell will probably increase. In connection with this development there is a strong demand on quality control of the products they want

to sell abroad. Customers of the industrilized countries have to be sure that the quality control is performed in accordance with international status.

The best way to fullfill these requirement is to carry out international standard tests for quality control. To assure the quality of these tests the general trend is a largely automatic procedure, which in particular means computer controlled tests. The development in the field of mechanical testing, in which the amount of standard tests is very high, shows the possibilities and tasks of the applied electronics and computers.

The same goal, higher quality assurance, is obtained by the further developments of tests, by which the properties are checked under conditions closer to the practice. In the field of mechanical properties examples are creep and fatigue tests, which even run under corrosive atmosphere now. Beside the field of nondestructive testing(it is not included in this report) in which tests are further developped and new ones are found, a rather recent method in materials testing is the application of acoustic emission.

A completely different trend is described with the thermophysical properties tests. Mechanical properties were always very important, therefore tests are established for a long time, which are continuously improved of course and completed, and the most important ones are an international accepted standard already. In thermophysical testing the properties became more important related to the energy situation and therefore thermal conductivity, specific heat, thermal expansion and so on have to be included in material characterisation to a much greater extend than in the past. In accordance with this trend some new methods were developped and the accuracy of the existing are carefully checked. In addition

there are a number of standardisation procedures going on.

Generally spoken the modern trend in quality control is to make the tests

- quicker
- more accurate
- more reproducible
- more reliable

With respect to the development of quality control in developing countries some recommendations may be made

- In general it is very useful to apply a personal computer to the test equipment. Experience have shown that a personal computer close to the test is better than a huge central computing unit.
- Every test should run automatically as far as possible.
- In a field where standard tests exist they should preferably used.
- For self-developed methods a routine procedure should be established and documented. In addition it is important to fix a quality assurance programm for the test.
- Related to the equipment, it should be pointed out, that in many cases the larger the possible field of application the more efficient the apparatus are. Examples are the comparative or laser-flash-method respectively for thermal conductivity measurements, tube dilatometers for thermal expansion and the differential scanning calorimeter for the determination of specific heat.