

April 2006

OnSet¹

News, Facts and Professional Solutions for Thermal Analysis

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The new NETZSCH homepage

The journey into the world of Thermal Analysis via internet has just become by far more attractive and informative. Our new homepage www.netzsch-thermal-analysis.com provides you with comprehensive information on our products and services, tailored and target-oriented to your line of business, your application, or your material.

With the first entry portal "Service/Support", we present our offering of a variety of comprehensive services for our customers not limited to just basic information. Select a technical division from "Technical Service - Applications - Measurements - Training & Seminars - Compliance - Applications Advisory Service - Literature Service" and clarify your questions - also directly by e-mail.

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Editorial

Continuation Page 1

The portal "Branches/Industries" grants you access via your industrial or applications field, e.g. "Polymer Manufacturing/Processing".

If your field of interest is focused on ceramic materials, for example, then simply go to page "Ceramic/Glass/Building Materials" via our portal "Materials/Applications". And if you are already familiar with the methods of Thermal Analysis, just click on our fourth portal "Products/Solutions". Here, diverse thermoanalytical measuring instruments, methods, and software products are summarized from which you can systematically select what you are interested in. By means of the individual categories "General - Technique - Software - Applications - Accessories", you gain access to detailed information.

The headline of our new homepage was entirely revised with a full-text search engine, interesting facts "About NETZSCH" and breaking news. In order for you to be quickly provided with the most essential things, we created the page "News" in "Highlights - Products - Applications - Events". Check it out for yourself! We look forward to receiving your requests and comments.

Dear Readers:

It is my great pleasure today to present you with the latest edition of our NETZSCH customers' magazine ONSET in 2006 - the "year of exhibitions". On the occasion of the large trade fairs Analytica 2006, Achema 2006 and Ceramitec 2006, a very interesting and newly designed edition covering the versatile field of "Thermal Analysis" with worth-reading articles on characterization of materials in the electronic, metal and polymer industries awaits you. With tips and tricks from renowned scientists, you will be familiarized in each issue with the key methods and know-how from decades of applications experience.

With quality as a matter of principle – as a worldwide recognized leader in Thermal Analysis, the team of experts from NETZSCH Analyzing and Testing will also be present this year at all important trade fairs. I cordially invite you to visit our new internet portal www.netzsch-thermal-analysis.com in advance.

We are looking forward to talking with you!

Now, please sit back and enjoy reading this issue of ONSET.

Sincerely yours,

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Stephan Knappe,
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DEA and Kinetic Analysis for Control of the Curing of Chip Adhesives

Dr. rer. nat. Harald Preu,
Infineon Technologies AG

Normally, end users do not ever come face to face with microelectronic parts with integrated circuits (IC). These are employed in PC circuit boards, in electronic entertainment devices, cellular phones and vehicular motor control units, where they dependably perform their services. To achieve functionality, however, the parts often undergo up to 500 processing steps, spanning from the production and structuring of silicon wafers to contacting, from recasting with reactive polymer compounds to soldering on the printed circuit board.

Electronic parts

For such a variety of individual processes, it is, of course, essential to minimize the error rate in order to guarantee cost-effective production. It goes without saying that the electronic parts must meet a multitude of reliability criteria. The parts for cellular phones, for example, must withstand the so-called drop test. The integrated component must withstand the stresses which may occur when a cellular phone is dropped. Concerning parts for automobile electronics, there are special demands regarding the resistance against moisture and temperature.

For these reasons, the materials employed and their production play a very specific role. Particularly polymer adhesives connecting the chip to the carrier material are under considerable stress in such a part since the connecting partners (silicon chip and substrate) differ widely in their thermomechanical properties (coefficient of thermal expansion, Young's modulus). Likewise important is, of course, fast processing of the adhesive, i.e. both the respective rheologi-

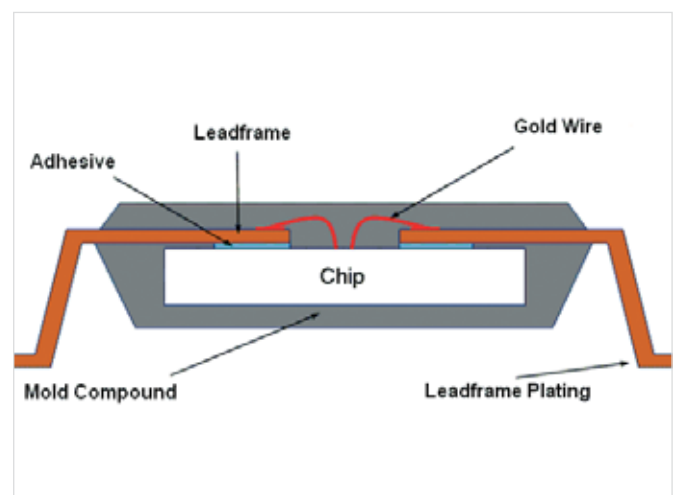


Infineon package for a controller

cal properties and optimum curing behavior must be guaranteed. The curing process is especially time-consuming and thus also influences the throughput and must be optimized.

Thermal Analysis as the ideal tool

Thermal Analysis offers ideal methods for this. Particularly by means of dielectric analysis (DEA) and kinetic analysis of the measured data, we



Typical Packaging. The chip is mounted onto the metallic leadframe by means of the adhesive; the electronic connection is made via gold wires.

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have had good research experience. For dielectric measurements in the form of pure cure monitoring, Infinion uses the DEA 231/1 *Epsilon* with a data acquisition time of up to 55 ms, necessary for fast curing systems. The following examples were all measured with IDEX S065 comb-shaped sensors. With the *Thermokinetics 2* software by NETZSCH, the measured data can be subjected to a formal kinetic analysis and predictions on the curing behavior at different temperature programs can then be made.

1. Curing of an adhesive with a temperature ramp of 23°C to 220°C in 123 s, approximately corresponding to process conditions (Fig. 1)

The so-called ion viscosity curve correlates with the dynamic viscosity of the material. The drop during the heating phase can be explained by a decreasing viscosity and therefore an increasing mobility of the charge carrier in the polymer. The signal mini-

mum denotes the point at which the signal, which was at first dominated by the softening of the material, begins being determined by the increasing curing. Curing of the adhesive is reflected by the increase in ion viscosity. The end of curing is determined when a constant value, here beginning at approx. 90 s, is achieved. A general topic in interpreting the ion viscosity process is the determination of the beginning and end of curing. The minimum was interpreted as the beginning of curing. The end, i.e. the entire curing, was determined by means of a tangent structure.

From the course of the ion viscosity curve, it can be concluded that the entire curing occurred during heating. This could mean for processing that curing can either be achieved at a lower temperature or at a shorter time. In the first case, this would mean a lower thermal stress for the component during production. In the second case, this would result in shorter processing times and thus, a

higher throughput.

2. Comparison of two measurements on the same material with an almost identical temperature ramp (Fig. 2)

Overlapping the curves shows the sensitivity of the measurements to a different temperature control. The ramp with a higher slope leads to earlier achievement of both the start



Curing of an adhesive

DEA 231/1 Epsilon with a data acquisition rate of up to 0.055s



(minimum) and the end of curing. The influence of the temperature is obvious. This is the strength of this method; the curing process is immediately visible and comparable.

3. Isothermal measurements at different temperatures and kinetic analysis (Fig. 3 + 4)

The measurements were carried out under isothermal conditions at 150, 170 and 200°C. The formal kinetic evaluation was made with the *Thermokinetics 2* program. The quality of the fit with a correlation coefficient of 0.9998 is excellent. Obviously, it is possible to describe

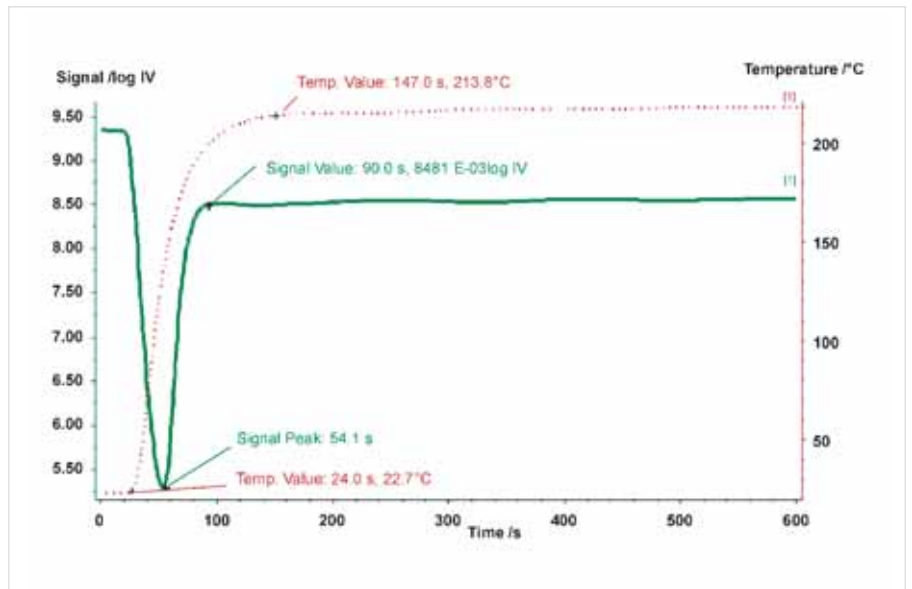


Fig. 1. Measurement of an adhesive during a temperature ramp.

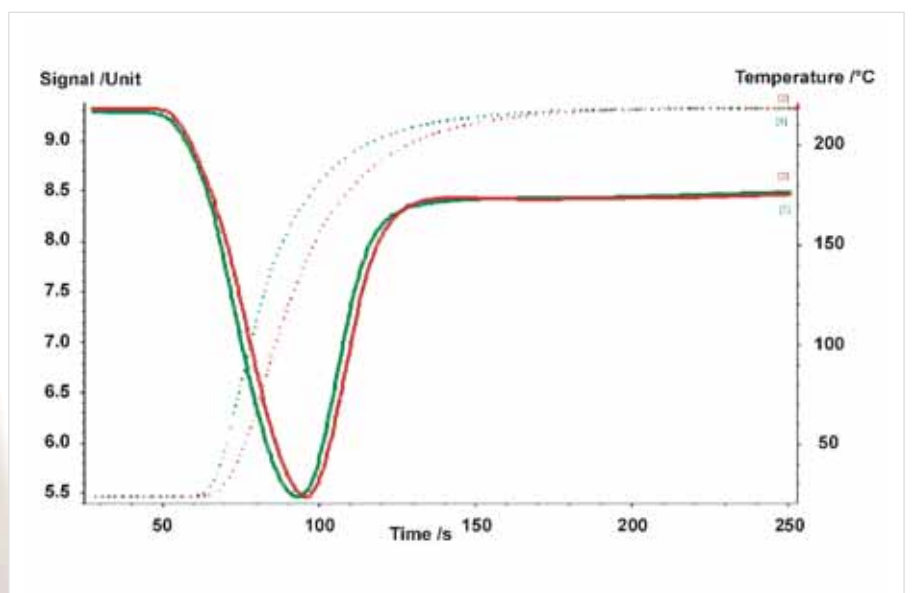


Fig 2. Comparison of two measurements at slightly different heating curves.

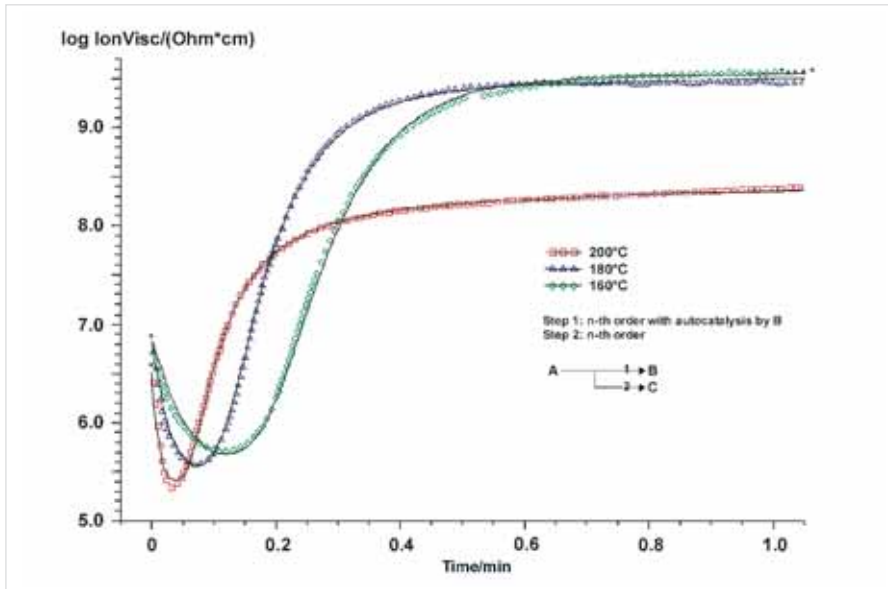


Fig. 3. Fit of a kinetic model to the experimental data.

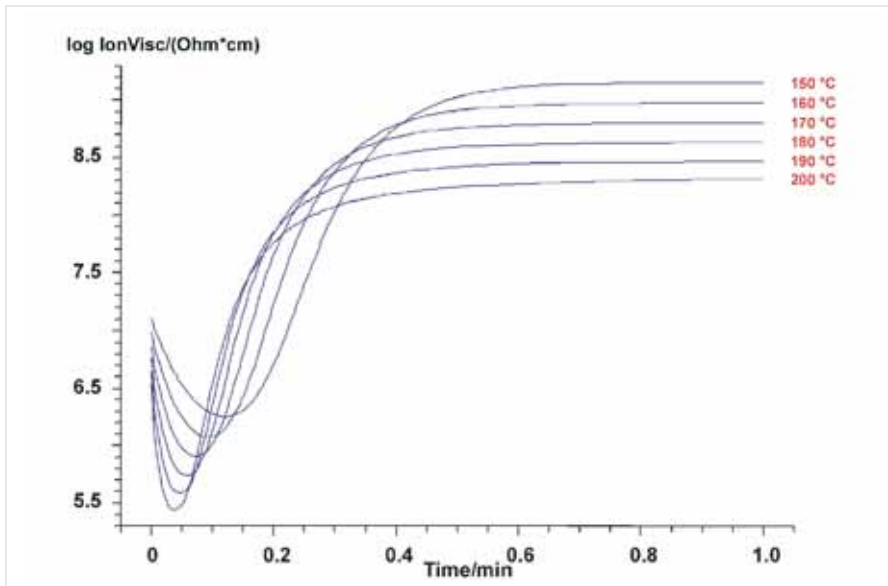


Fig. 4. Calculation of ion viscosity curves with the help of kinetic model resulting from the fit to the experimental data. Isothermal conditions were assumed.

the experimental data with one parameter set. A double-step reaction was employed as a reaction model, where the 1st step was of n -th order with autocatalysis and the 2nd step was of n -th order. A variety of other kinetic models can be selected.

It can clearly be seen from the course of the ion viscosity curves that curing – dependent on the temperature – occurs in a timeframe of approx. 12 – 25 s. Such a fast reaction can hardly be characterized by means of rheology. With the help of the kinetic analysis, the course of curing reactions can now be determined at almost any temperature.

In this case, isothermal conditions were simulated. The numeric data can be stored as ASCII files from the *Thermokinetics 2* software and are available for further evaluation. They can now be used to determine, for example, curing time as a function of temperature (Fig. 5).

As expected, the biggest influence of the temperature change on the curing time can be observed at lower temperatures. At higher temperatures, curing cannot be arbitrarily shortened. This makes it possible to carry out a fast and particularly clear process optimization. Further, the comparison of different adhesives is facilitated and thereby the selection of materials as well.

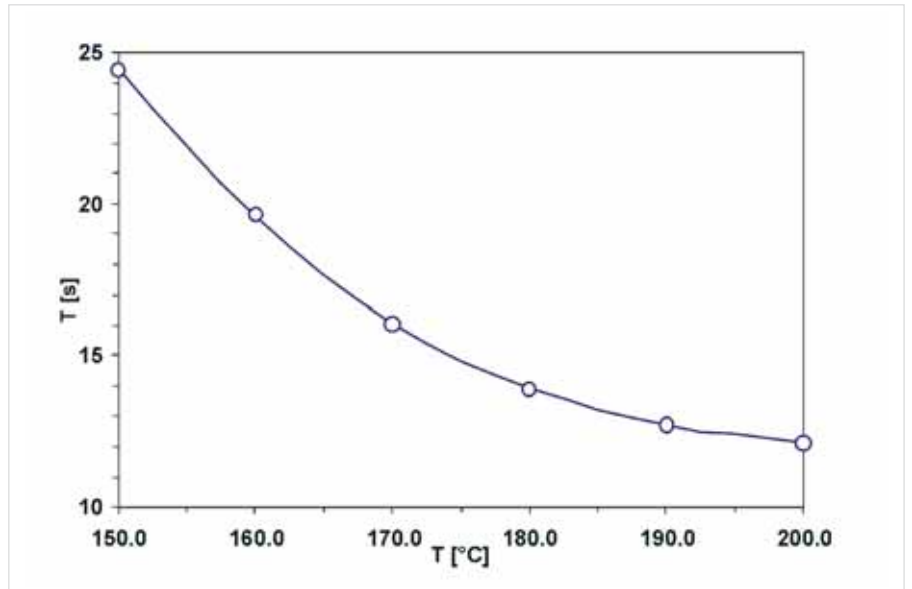


Fig. 5. Application of curing times as a function of temperature. The values were extracted from the simulated curves.



The Author:

Since 2001, Dr. rer. nat. Harald Preu has been employed at Infineon Technologies AG in Regensburg, Germany, in the field of package development. His main work concentrates on polymer analysis, especially by using thermal analysis methods.

Infineon in few words:

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Infineon's know-how is used in automobiles, computers and telecommunications. It is as important in the fields of safety and integrated circuit boards as it is in industrial electronics and health care. The product portfolio includes integrated circuits (ICs), storage and logic products as well as discrete semi-conductor products.

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Never stop thinking

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Heat Transfer Analysis in Layer Structures

Dr. Jürgen Blumm
NETZSCH Applications Laboratory

Characterization of the heat transfer in multi-layer systems is getting more and more important in a wide range of industrial applications. A famous example is the analysis of the heat transfer in electronic components (electronic packaging). Increasing the efficiency of the heat transfer out of the active electronic component allows higher clock speeds without the risk of thermally induced damage. Another example of a multi-layer system is the thermal barrier coating which is used more and more frequently in the field of high-temperature gas turbines. Such coatings protect the metal substrate from the corrosive gases in the gas turbine. Furthermore, due to the low thermal conductivity of such ceramic coatings, a significant temperature gradient can be established over the thickness of the coating, allowing the gas turbine to work at higher temperatures. The system's efficiency can therefore be increased.

The laser flash method

For decades, the laser flash method [1] has been well-known for characterizing the thermophysical properties of thin solid materials. In a laser flash test, the front side of a plan-parallel sample disk is heated by a short laser pulse. The heat diffuses through the sample and leads to a temperature rise on the rear side of the sample. By measuring this temperature rise versus time, the thermal diffusivity of the sample can be determined. Fast measurement times, easy sample preparation, and high accuracy are only some of the advantages of this non-contact, non-destructive measurement technique. Furthermore, the method can easily be adapted to the

analysis of multi-layer samples. The principle sample setup in a two/three layer analysis in a flash measurement are presented in figures 1 and 2.

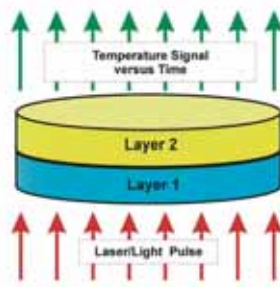


Fig. 1. Schematic setup of a two-layer test in a flash device

One problem in multi-layer analysis using the laser flash method is analysis of the measured data. Most analytical models for description of the transient heat transfer during a laser flash test are based on an adiabatic model. This means that any heat loss effects from the sample surface are not taken into account in the analysis.

Theoretical model

Solutions for the analysis of multi-layer systems (two- and three-layer systems) were presented as early as 1975 by Lee [2]. For consideration of heat loss effects, a procedure developed for single-layer systems by Cowan [3] can be used for multi-layer systems.

$$T(d_2, t)_{\text{heat loss}} = T(d_2, t)_{\text{adiabatic}} \cdot \exp\left(-\frac{\beta t}{(d_1 + d_2)^2}\right)$$

New analysis routine

The new analysis routine is integrated into a non-linear regression routine

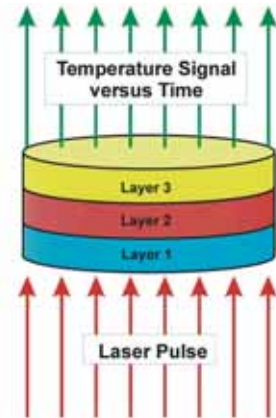


Fig. 2. Schematic setup of a three-layer test in a flash device

[4], allowing a curve fit of the measurement results of laser flash experiments. An example of a fit of a measurement on a two-layer system (thermal barrier coating on an Ni-



LFA 427

based superalloy substrate) is presented in figure 3.

It can clearly be seen that the new model can describe the measurement result in an accurate way. Tests on standard materials such as artificial two-layer systems or two-layer systems made of two standard materials have proven the reliability of the new method. The uncertainty of the new model is within $\pm 5\%$ as long as the unknown layer has a significant contribution ($>50\%$) to the thermal resistance of the layer system. Of course, it has to be pointed out that for the analysis of the two-layer system, the thermo-physical properties (specific heat, density and thermal diffusivity) of one layer must be known.

Furthermore, the specific heat and density of the second layer must also

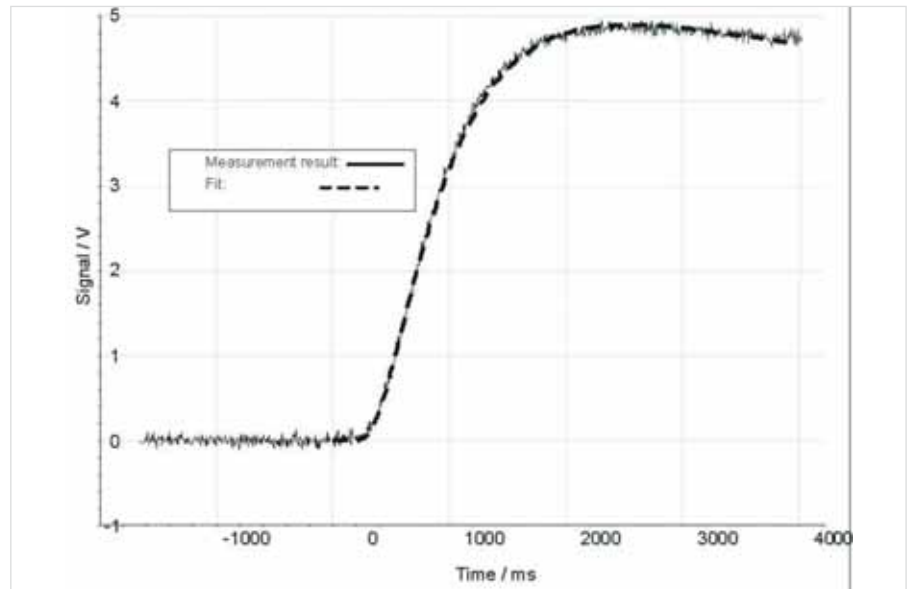


Fig. 3. Comparison of a measurement result on a two-layer system and the corresponding fit.

be known. The same applies to three-layer systems.

Results and Discussion

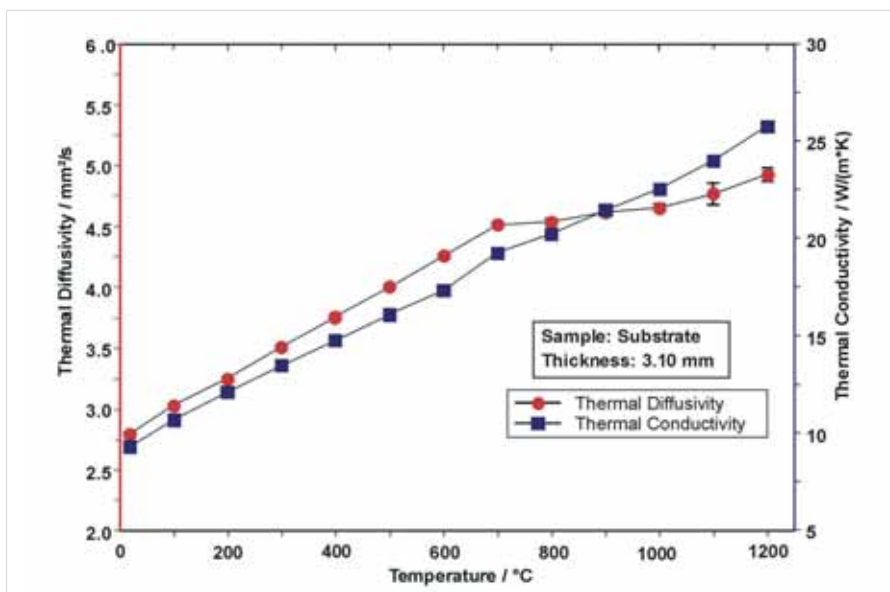


Fig. 4. Thermal diffusivity and thermal conductivity of an Ni-based superalloy.

Presented in figure 4 is a measurement result for the thermal diffusivity and thermal conductivity of an Ni-based superalloy used as a substrate for thermal barrier coatings in the gas turbine industry. It can clearly be seen that both the thermal diffusivity and thermal conductivity of the Ni-based superalloy increase with temperature. Above 700°C , the increase in thermal diffusivity changes. This effect is due to a phase transition in the alloy. Due to a significant increase in the specific heat (not shown in the plot) in the same temperature range, the increase in the resulting thermal conductivity is nearly linear.

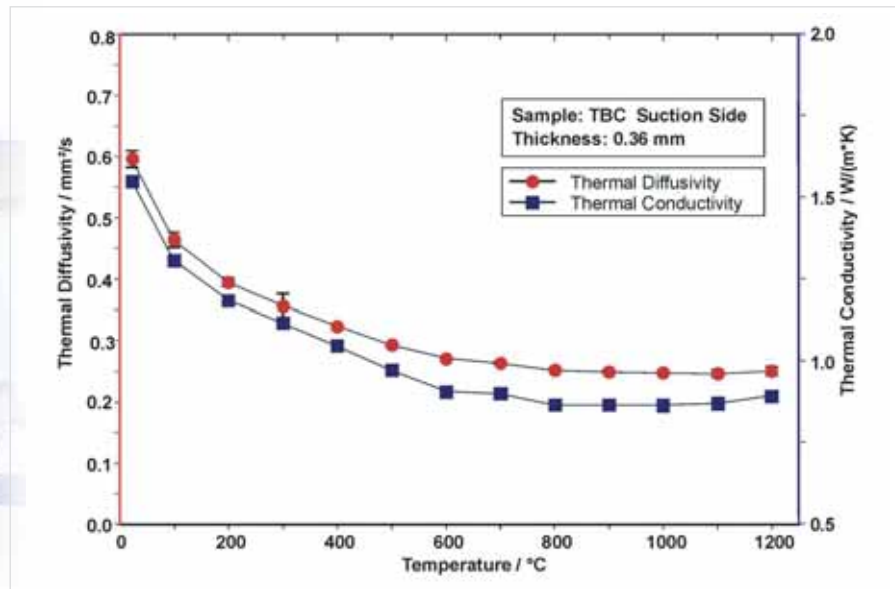


Fig. 5. Thermal diffusivity and thermal conductivity of a zirconia thermal barrier coating (suction side of the turbine blade) measured in a two-layer system.

Shown in figure 5 are the thermal diffusivity and thermal conductivity of an yttria-stabilized zirconia thermal barrier coating between room temperature and 1200°C. The measurement was carried out on a two-layer system with the thermal barrier coating plasma-sprayed on the substrate material. The system analyzed here had already been used in a gas turbine system for 20,000 hours prior to the measurement. The sample was taken from the suction side of the turbine blade. It can clearly be seen that the thermal diffusivity and thermal conductivity at room temperature are in the typical range for porous stabilized zirconia materials (porosity approx. 12%). Furthermore, the decrease of both thermophysical properties versus temperature is typical for the behavior of ceramic materials.

Depicted in figure 6 are the measurement results for a sample taken from

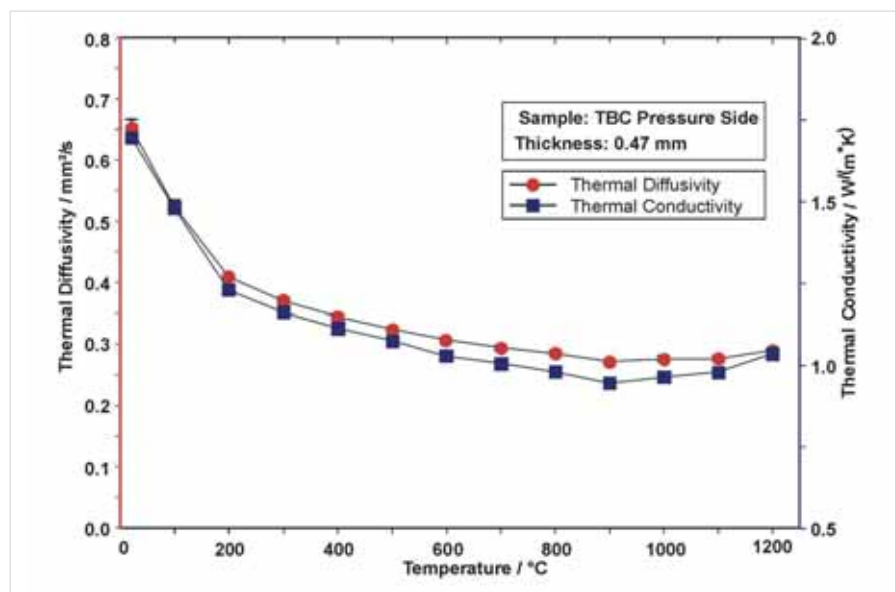


Fig. 6. Thermal diffusivity and thermal conductivity of a zirconia thermal barrier coating (pressure side of the turbine blade) measured in a two-layer system.

the pressure side of the turbine blade. It can clearly be seen that both the thermal diffusivity and thermal conductivity are significantly higher than the results for the sample taken from the suction side of the turbine

blade. This is most probably due to the different thermal and mechanical treatment of the different sides of the turbine blade during use.

Presented in figure 7 is an example of measurement results employing a three-layer analysis. The figure shows the thermal diffusivity and thermal conductivity of water measured in an aluminum container for liquids, where the sample is positioned between two aluminum plates with a well-defined thickness. The tests were carried out between 25 and 50°C. Literature values [5] for density and specific heat were employed for the analysis. Additionally, literature values for the thermal conductivity of water are given. It can clearly be seen that the results for the thermal conductivity are in the typical range for water. The difference between the individual results and literature values are less than $\pm 2\%$. This reproducibility and low level of uncertainty were obtained over the entire temperature range.

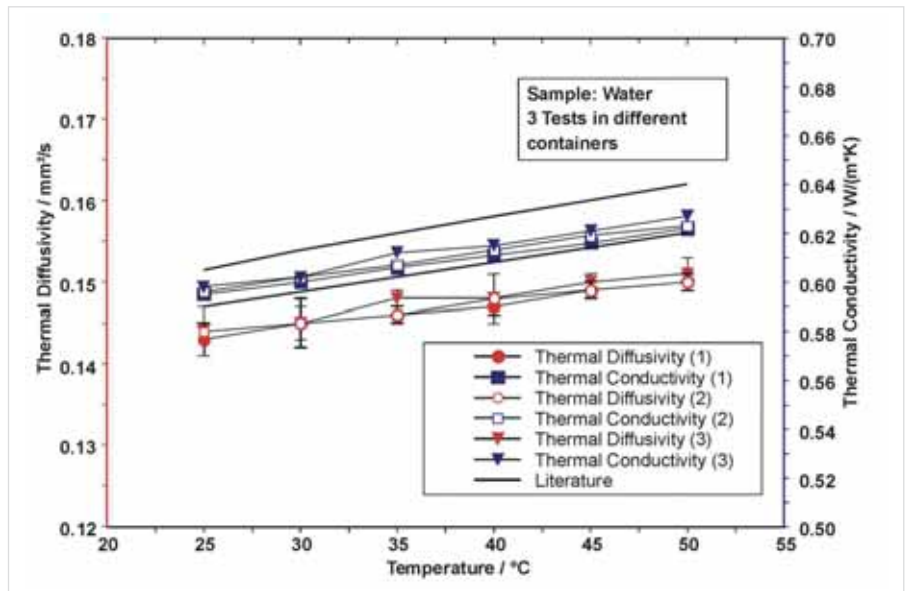


Fig. 7. Thermal diffusivity and thermal conductivity of water measured in an aluminum container (three-layer analysis)

References

[1] Parker W J, Jenkins R J, Butler C P, Abbott G L, 1961 J. Appl. Phys., Vol. 32, 1679-1684

[2] Lee H J, 1975 PHD-Thesis, Purdue University
 [3] Cowan R D, 1963 J. Appl. Phys., Vol. 34, 926-929
 [4] Opfermann J R, 1985 Rechentechnik/
 Datenverarbeitung 22, 3, 26-27

[5] Incropera F P, DeWitt D P, Introduction to Heat Transfer, John Wiley & Sons, Inc. 1996

Not Even Platinum is Everlasting!

Dr. Gabriele Kaiser
 Training

Thermocouples have become established as standard temperature measuring devices in thermal analysis: they feature simple set-up and operation, and they are multifunctional, robust and compact.

The most frequently used thermocouple material for operation above



800°C is platinum-platinum/rhodium (10%) – with regard to its chemical composition, also designated Pt-Pt10%Rh,- or also referred to as type S. The main advantages of this thermocouple, developed by Le Chetelier more than 100 years ago, are high reproducibility, good corrosion and oxidative stability.

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Set-up:

The negative side of the thermocouple consists of platinum; the positive – in accordance with ASTM E 1159 – of platinum/rhodium with a weight proportion of approx. 10.00 +/- 0.05% rhodium (see picture 1).

Resistance:

Compact platinum–platinum/rhodium features a virtually unlimited resistance at room temperature. This, however, changes under regular operation at high temperatures. Interdiffusion, selective evaporation, recrystallization and environmental influences are the main reasons for changes in thermal tension or failure of the thermocouple.

a) Selective evaporation and interdiffusion

At temperatures above 1000°C, evaporation of rhodium and also diffusion of rhodium from the positive Pt10%Rh side to the negative Pt side occur. Both effects result in impurities and increased wear and tear of the platinum wire.

In order to minimize the risk of alloy formation over the gas phase, the majority of the thermocouple wire for DSC/DTA sample carriers is protected by a capillary of high-purity Al_2O_3 .

b) Recrystallization

In the temperature range above 1100°C, platinum recrystallizes into a coarse-grained structure (see picture 2).

The described grain growth not only occurs within the metal or metal alloy, but also leads to “coalescence” of different platinum parts which

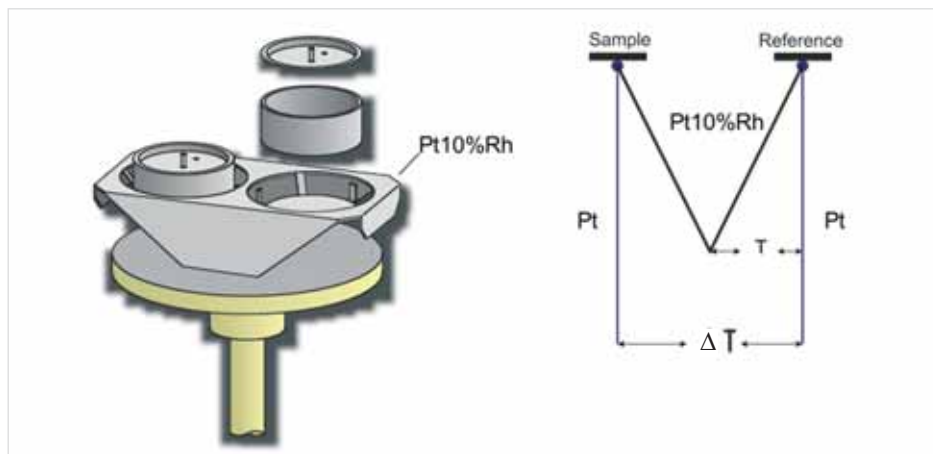


Fig. 2. DSC Sample Holder

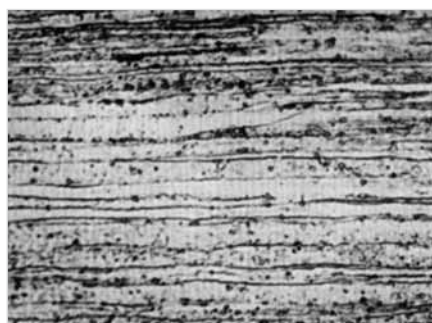


Fig. 2a. Structure of a rolled instrument platinum wire* (magnification 400 :1)

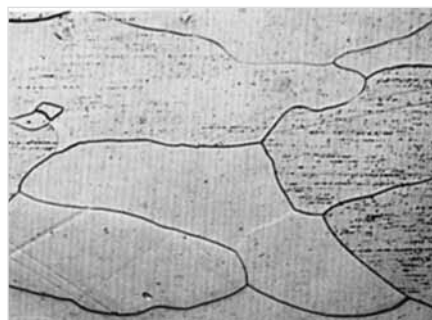


Fig. 2b. Thermo platinum wire, 30 min at 1200°C* (magnification 100:1)

are in contact with each other, such as DSC/TG sensor type S and Pt/Rh DSC crucibles. Only conditioning of new sample carriers and crucibles by means of a special thermal treatment lowers the “sticking tendency”.

Using unconditioned sample carriers and crucibles in the temperature range above 1000°C immediately leads to welding of the crucible onto the sensor and thus, destruction of the sample carrier.

Please pay attention to the instruction leaflet of your sample carrier in this regard. We would ask you to heat new Pt/Rh crucibles prior to using them in a separate furnace to the required end temperature of the measurement, lift the crucibles from the sensor after each measurement as a precaution, and go up to temperatures above 1100°C only step-wise in the beginning.

In our experience, using dispersion-hardened (so-called FKS) materials for the sensor surfaces and crucibles does not result in a long-term significant improvement.

One way of avoiding the described phenomenon is to underlay thin sapphire disks (between sensor surface and crucible). The risk of sticking is minimized and the sensitivity of the sample carrier is only slightly decreased.



Fig. 3. DSC sample carrier type S

c) Environmental effects

In practice, the biggest influence on the service life of thermocouples is due to interactions with the environment. Diffused impurities, released from the samples, change the thermal tension or may even cause initial cracking of the thermocouple wire. In the table, you will find details on the chemical compatibility of platinum with other sample materials and gas atmospheres.

This list demonstrates how important regular inspections and calibration measurements are. This is the only way to ensure that the employed thermocouple material Pt-Pt10%Rh does not exceed the defined tolerance limit over a longer period of time.

Critical for platinum:

- Halogens (Cl_2 , F_2 , Br_2), aqua regia
- Li_2CO_3 , prior to emission of CO_2
- PBO, FeCl_3
- Be alloys (evaporation starts shortly above the melting point)
- HCl with oxidants (e.g. chromic acid, manganates, iron(III)-salts, molten salts)
- reducing atmospheres
- P, B, metal and metal vapors such as B, Pb, Zn, Sn, Ag, Au, Li, Na, K, Sb, Bi, Ni, Fe, steel, As, Si
- Selenium above 320°C (immediate cooling and removal of the sample at the end of the measurement recommended to prevent evaporation of selenium)
- Metal oxides with reducing substances such as C, organic compounds or H_2
- Oxides in an inert gas atmosphere at higher temperatures (reduction)
- Sulfur (roughening of the surface, embrittlement)
- Alkali hydroxides, alkali carbonates, alkali sulfates, alkali cyanides and rhodanides at higher temperatures
- KHSO_4 at higher temperatures
- Carbon black or free carbon above 1000°C
- SiO_2 under reducing conditions
- SiC and Si_3N_4 above 1000°C (release of elementary Si)
- HBr, KC solution at higher temperatures

No resistance to:

- Mixtures of KNO_3 and NaOH at 700°C under exclusion of air
- Mixtures of KOH and K_2S at 700°C under exclusion of air
- LiCl at 600°C
- MgCl_2 , $\text{Ba}(\text{NO}_3)_2$ at 700°C
- HBr, HJ, H_2O_2 (30%) and HNO_3 at 100°C
- KCl (the decomposition products which form during melting; melting point: 768°C)

Limited resistance to:

- KHF_2 , LiF_2 , NaCl at 900°C
- Mixtures of NaOH and NaNO_3 at 700°C under exclusion of air

No claim is made that this overview is exhaustive; it is only a guideline for the user. For the most part, the temperatures are literature values. The temperatures under test conditions might be shifted to lower values. It is always advisable to run preliminary tests in separate furnaces. NETZSCH-Gerätebau excludes liability for any damages resulting from improper use of the instruments, crucibles, sample carriers, etc.

* Lit: Edelmetall-Taschenbuch, Degussa AG/Frankfurt, 1967
Lit: Edelmetall-Taschenbuch, Degussa AG/Frankfurt, 1967

World-wide Service in Thermal Analysis

Andreas Strobel
Technical Service

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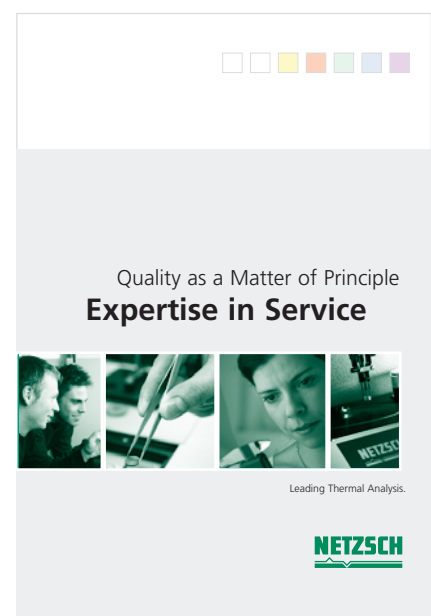


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A more detailed description of our



Meeting Point Fair

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Training



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- from April 25th – 28th, 2006 at Analytica 2006 in Munich, hall A1, booth 305,
- from May 15th – 19th, 2006 at Achema 2006 in Frankfurt, hall 6.3, booth H 23
- from May 16th - 19th, 2006 at Ceramitec 2006 in Munich, booth B6.301.

You are cordially invited!

Benefit from our know-how

At Analytica, the world's largest trade fair for analysis, laboratory techno-

logy and life science, NETZSCH Analyzing & Testing will be hosting two seminars on April 27th, 2006 in room A11 in addition to our presence at the trade fair. The morning of April 27th, starting at 10:30, is dedicated to "Application of Thermal Analysis in the Polymer Field"; the afternoon, starting at 13:35, is focused on "Thermal Analysis in the Pharmaceutical Industry". You are cordially invited to attend these two events. For more details and the corresponding registration forms, please visit our homepage www.netzsch-thermal-analysis.com under *News/Events*.

We are your ideal partner for the laboratory

At ACHEMA 2006, the leading international trade fair for suppliers of the chemical industry, laboratory and analysis technology represents the second largest exhibiting group. NETZSCH Analyzing & Testing is presented with a broad range of instruments and service for Thermal Analysis. This enables you to be

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Our Events:

Event	Date	Location
Analytica 2006	25. - 28.04.	Munich
Achema 2006	15. - 19.05.	Frankfurt
Ceramitec 2006	16. - 19.05.	Munich
29 SBQ Soc Bras Quimica	19. - 22.05.	Aguas de Lindoia
International Symposium on Inorganic Interfacial Engineering	20. - 21.06.	Stockholm
Rohima 2006	27. - 28.06.	Leipzig
22 nd European Symposium on Applied Thermodynamics	28.06. - 01.07.	Copenhagen
9 th European Symposium on Thermal Analysis and Calorimetry	27. - 31.08.	Cracow
Nano Korea 2006	30.08. - 01.09.	Goyang
PhandTA9	10. - 13.09.	Düsseldorf
Analytica China 2006	19. - 21.09.	Shanghai
Tecnargilla 2006	28.09. - 02.10.	Rimini
IMAPS Electronic Packaging Conference	08. - 12.10.	San Diego
Zwick Fachmesse	16. - 19.10.	Ulm
FAKUMA	17. - 21.10.	Friedrichshafen
MS&T, Materials Science & Technology	17. - 18.10	Cincinnati

It's Quiz Time



Find out the answer! It is a term from standardization.

Visit our corresponding booth at the coming trade fairs and get your price.

- In which room are the two NETZSCH seminars taking place during Analytica?
- Which measuring method is employed at Infenion (7th letter)?
- What is the technical term (letter) for the thermocouple developed by LeChatelier more than 100 years ago?
- The 11th letter of our new website is?

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