onset

Thermal Analysis

March 2004

The Formula 1 among Thermobalances

Figure 1. The new TG 209 **F1** Iris®

After the successful launch of our DSC 204 *F1 Phoenix*®, we are again in the pole position with our new TG 209 *F1 Iris*®. The vacuum-tight thermomicrobalance, designed for the temperature range from 10°C to 1000°C, features a functional design with integrated components which are controlled by the *Proteus*® software: AutoVac for automatic evacuation and gas filling and three mass flow control units for the exact regulation of the purge and protective gases. The system can be upgraded with an automatic sample changer (ASC) for up to 64 different crucibles, including an optional piercing device for the crucible lids. The ASC can also be mounted on the DSC 204 *F1*. Simultaneous coupling of the TG 209 *F1* to an FTIR and to the QMS 403 *Aëolos*® via a new, heatable adapter system, enables a gas analysis even when the ASC is being used.

Figure 2 shows the TG and DTG curves for the decomposition of an NR/SBR rubber mixture up to 550°C in nitrogen. The atmosphere was then changed to air in order to generate the combustion of the added carbon black and to determine the ash content (residual mass). The illustration of the protective and purge gases employed confirms the exact regulation of ±0.5 ml/min.

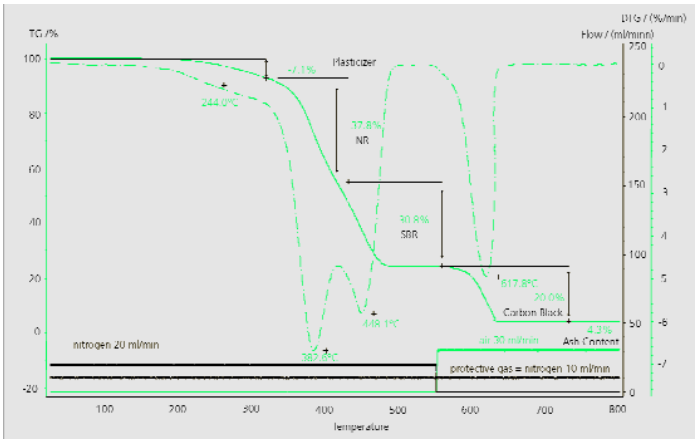


Figure 2. Composition of an NR/SBR Rubber Mixture

Software Questions?

Dr. Elena Mouhkina

Since September 2002, Dr. Elena Moukhina, 37, has dedicated herself to the further development of the "Advanced Software". Since that time, she has succeeded in transferring the *Thermal Simulation*, *Peak Separation*, *Purity Determination*, *Density* and *DSC/DTA Correction* programs from the 16-bit into the 32-bit structure and extending some important functions.

Before Dr. Moukhina joined NGB in 2002, she worked for nine years as an associate university professor for information technology at the University of Jaroslawl, Russia.

If you have any questions regarding the Advanced Software - including *Thermokinetics* or *Chem Rheo*® - do not hesitate to contact Dr. Moukhina at e.moukhina@ngb.netzsch.com. She is looking forward to hearing from you and will be happy to support you in any way she can.

by the way

... whether you are a beginner or already an experienced user of Thermal Analysis, our seminars in Selb offer the right platform for discussing calibration, sample preparation, measuring conditions and curve interpretation and learning about new features in hardware and software. More details on our courses in English can be found on our homepage www.ngb.netzsch.com.

We look forward to welcoming you in Selb!

Contents

NGB Extends the DMA 242 C *Proteus*® Software

For more than one year, the DMA software has been part of the *Proteus*® software group. In Version 4.6, available now, not only have the automatically running calibration routines been extended for the wide variety of different parameters such as sample holder type, force and amplitude, frequency, temperature and time, but parameter input and presentation of curves in evaluation have also been facilitated for the user.

Static measuring modes like "relaxation" and "creep" as well as "TMA" for determination of the thermal expansion coefficient have been completely revised. Here, polymeric materials are investigated in the compression/penetration or tensile mode. Additionally to these modes, the dynamic deformations "stress and strain sweep" can be directly selected at the input of the data file and can then be evaluated in a separate window. In "stress sweep", a series of different force amplitudes is applied to the sample and the effects on the sample's length or thickness at a certain temperature are determined as a function of time. In "strain sweep", especially interesting for rubber samples, it is just the opposite: a sequence of different deformation amplitudes is applied as cycles. Here, the dynamic force required for the sample deformation is measured as a function of time in an isothermal phase.

Ask for an upgrade of your existing 16-bit version for the DMA 242 or DMA 242 C! Besides the software upgrade, you only have to install the new EEPROM in your DMA controller. The new *Proteus*® software of the DMA runs under Windows 2000 or Windows XP Professional.

Key Features of the Measuring and Evaluation Software

- Multitasking for the simultaneous operation with up to 4 instruments
- Multiple temperature segments and ramps with freely selectable parameters programmable
- On-line graphic with up to 5 individually selectable signals and axes, time- or temperature-scaled
- PIP (picture in picture) to show zoomed graphic sections and to exchange entire graphics and sections (FLIP)
- Snapshot: On-line evaluation of the measurement in progress
- Evaluations in multiple window technique
- Storage of the analysis results and graphic
- Export of graphics as bitmap or enhanced metafile format
- Revised context-sensitive help system with many practical tips integrated now

NETZSCH GEFTA Prize 2003 Goes to Dr. Dr. Jens Fischer

Dr. Dr. Jens Fischer (in the middle) with Dr. Michael Feist (GEFTA chairman, left) and Thomas Rampke (Sales Manager Germany at NGB,right).

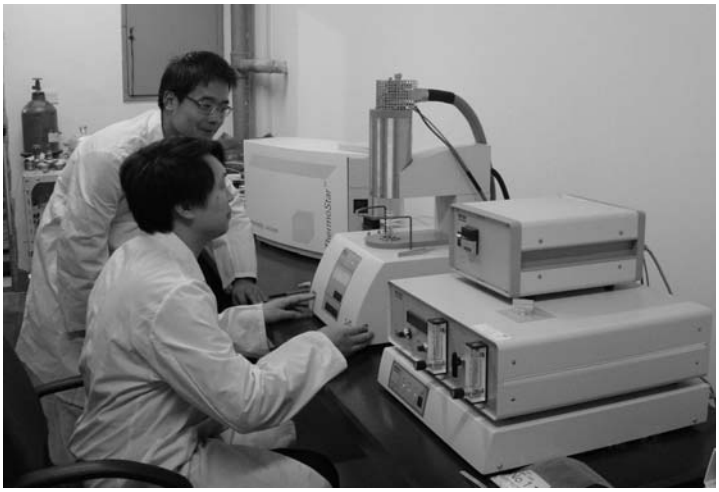
Since 1977, the NETZSCH GEFTA Prize, to which 2,000 euros are currently allocated, is awarded in honor of outstanding achievements in the field of Thermal Analysis.

In 2003, it went to Dr. Dr. Jens Fischer, private lecturer and vice-chairman of the hospital for dental prosthetics at the University in Bern. The ceremonial awards show took place on the occasion of the annual GEFTA meeting at the University of Augsburg last September.

Dr. Dr. Jens Fischer, dentist and crystallographer, carries out research in the field of dental materials and their processing technology. He has thereby succeeded in establishing a method for the quantitative determination of the burning stability of dental alloys, suited for routine investigations. By modifying a dilatometer, it became possible to measure the properties of test bodies under load (three-point bending) as a function of temperature. Based on these results, a palladium-free (and therefore hypoallergenic) titanium alloy containing high levels of gold could be developed, featuring a considerably higher burning stability than common noble metal alloys.

We cordially congratulate Dr. Dr. Fischer on this award and wish him much success for all his future projects.

Nanotechnology and Thermal Analysis



Prof. Weijie Ji and Mr. Qin Yu (in front) in front of their STA 449 *Jupiter*®-QMS combination

Nanjing University, China, houses a number of state and province key laboratories. One of them, the key laboratory for mesoscopic chemistry (director: Prof Zeng Hu), hosted by the School of Chemistry and Chemical Engineering, has been successfully working with NETZSCH instruments - a combination of an STA 449 C *Jupiter*® and a quadrupole mass spectrometer (see photo) - for approximately two years. Several professors of this lab, together with their post-doctoral fellow and graduated students, focused on the study of preparation, structures and properties of mesosopic materials.

The term "mesoscopy" (from Greek "meso", meaning "middle" and "skopos", meaning "purpose" or "attention") originates from physics and designates systems whose properties are a function of size (Kirk and Reed, 1992). In chemistry and crystallography, "mesoscopic" describes a range of sizes that lies between the microscopic and macroscopic scale.

This also includes fullerenes in the form of nano or bucky tubes, which are tiny carbon tubes with a length of up to 1 mm and a diameter of approximately 1 to 30 nm. Compared to previously known construction materials, they have unique properties such as considerably higher stability and stiffness or unusually high electric and thermal conductivity in connection with very good temperature stability. After the accidental discovery of carbon nanotubes (CNTs) by Japanese scientists in 1991, it is today possible to manufacture these on a gram scale. A frequently employed synthesis method is the Chemical Vapor Deposition (CVD) technique, where hydrocarbons (precursors) or carbon monoxide, among others, can be used as a carbon source. The chemical details of the growth process, however, are not yet entirely understood.

By means of their in-situ investigation of nanotube growth, the team of Prof. Hu and Prof. Ji has made a considerable contribution to the improved understanding of this mechanism. Benzene as a carbon source (nitrogen as a carrier gas for the benzene vapor) was brought into contact with an Fe-Co/y-Al_2O_3 catalyst (weight: approx. 3 mg) in an STA 449 C *Jupiter*® in the temperature range between room temperature and 700°C. The gases evolved during the reaction were analyzed on-line with a mass spectrometer (see picture) coupled via a capillary. It can be concluded by comparison of the TG and DSC data with the recorded spectra that, at a temperature above 645°C (the temperature at which the reaction begins), first the catalyst is reduced and then the dehydrogenated benzene on the catalyst surface is directly assembled into the carbon nanotubes.

The in-situ technique provides an effective way of understanding the growth mechanism also when using other precursor gases.

More information on the experiment described can be found on the website of the American Chemical Society *http://pubs3.acs.org/acs/journals/doilookup?in_doi=10.1021/ja037561l*

Prof. Zeng Hu

We thank Prof. Zeng Hu and Prof. Weijie Ji for providing us with this contribution.

Reinforcement in Sales/Marketing

Oliver Zeeb

As of January 2004, Oliver Zeeb, 39 - industrial and production engineer - has taken on the responsibility for "Sales Europe" and "Marketing" at NETZSCH-Gerätebau. He was previously employed in various corporate groups and medium-sized companies.

"I am convinced that with our products and with the support of our laboratory, we are the right problem-solving partner for our customers," replied Mr. Zeeb in answer to the question as to where our strength lies for our industrial and academic customers.

Take advantage of this strength and contact Mr. Zeeb or his team at *o.zeeb@ngb.netzsch.com*.

Trade Fairs, Symposia 04

We will be participating in the following exhibitions:

Forum Lab	23 - 26 March, Paris, France
Analytika Expo	5 - 8 April, Moscow, Russia
CBRATEC Brazilian Thermal Analysis Society	11 - 15 April, Pocos de Caldas, Brazil
Teheran International Conference of Refractories	4 - 6 May, Teheran, Iran
Analytica 2004	11 - 14 May, Munich, Germany
AIMAT 04	22 - 25 June, Rimini, Italy
Chinaplas	29 June - 3 July, Shanghai, China
Analytica China	7 - 9 September, Shanghai, China
ICTAC 13	12 - 18 September, Chia Laguna, Sardinia
Physique 2004	19 - 21 September, Paris, France
PhandTA 8	26 - 30 September, Monte Verita, Switzerland

New Co-Workers in the Lab

Markus Meyer

Markus Meyer was born in Selbitz, Upper Franconia, in 1972. Initially, he studied chemistry and biology to become an educator at high schools. After completing his diploma thesis in botany and the state examination, he focused on chemistry.

In October 2003, he started working in our applications laboratory, with an emphasis on DMA and Dilatometry. In the future, Mr. Meyer will also take over the responsibility for DEA.

Dr. André Lindemannn

Dr. André Lindemann was born in Wernigerode, Germany, in1971. He studied thermal engineering with an emphasis on process engineering at the Otto-von-Guericke University in Magdeburg, where he completed his doctorate in the field of simulation of thermal processes in technical thermodynamics.

As of November 2003, Dr. André Lindemann has been employed in the applications laboratory with a focus on the LFA product group.

In Memory of Dr. Johannes Opfermann

Dr. Johannes Opfermann

We mourn the loss of Dr. Johannes Opfermann, who passed away on January 23rd, following a brief, serious illness.

The dedicated scientist gained an international reputation with the development of programs for in-depth analysis of thermoanalytical data such as the *Peak Separation*, *Thermokinetics* or *ChemRheo*® software packages.

As early as the 70s, he began to focus on reaction kinetics, a subject he never laid to rest. And it was "kinetics" that brought him in contact with NETZSCH-Gerätebau in the middle of the 80s.
In the summer of 1991, Dr. Johannes Opfermann joined NGB as head of the software department. His wealth of ideas did not end with the NETZSCH *Proteus*® Software, however. From 1998 through 2002, he dedicated himself intensely to the "Advanced Software" and provided active support in both sales and applications. In 2002, he took over as head of the R&D department, a position which he held until his death.

In Dr. Opfermann, NGB has lost not only an extremely competent and engaged physicist, but also a warm-hearted and treasured colleague.

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