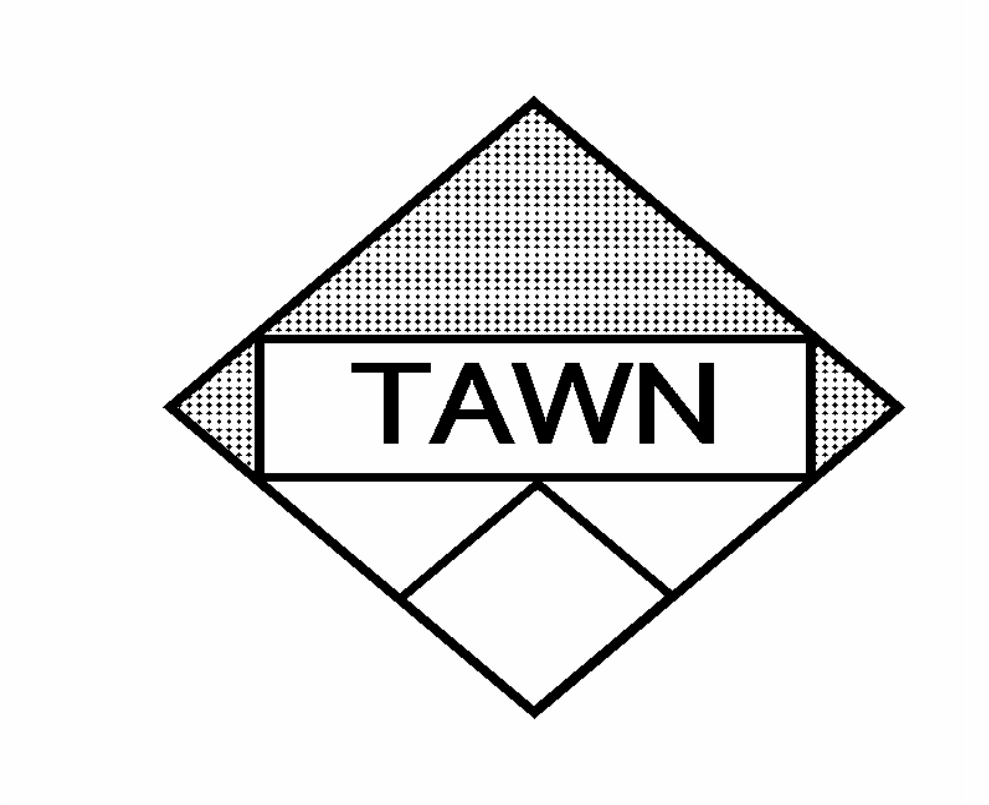
Thermische Analyse Bulletin

Het officiële orgaan van de Thermische Analyse Werkgroep Nederland sept. 2009



Conferentie data,
ESTAC 2010 , TAD 2009, Nomenclature,
Apparatuur, Wetenschappelijke bijdragen, Etc

Conferentie locatie de Doelen.
22-28 augustus 2010

COLOFON

Het Thermische Analyse Bulletin is het officiële orgaan van de Thermische Analyse Werkgroep Nederland (TAWN).
Het bulletin wordt gratis aan de leden gestuurd.

In het bulletin worden opgenomen:
- **nieuws van het bestuur van de werkgroep;**
- **gegevens over congressen, symposia en cursussen;**
- **internationaal nieuws;**
- **boekbesprekingen;**
- **gegevens over nieuwe apparatuur en de toepassing ervan.**

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REDACTIONEEL

De bijdragen van firma's en correspondenten wordt steeds minder waardoor het uitbrengen van een nieuw bulletin meer tijd kost. In dit bulletin de aankondiging voor de TAD 2009. Graag snel Uw bijdragen bekend maken zodat we nog voor de TAD een bulletin met het volledige programma kunnen presenteren.

Bestuur TAWN

Dr. P.J. van Ekeren, voorzitter

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Inhoudsopgave

1. Informatie en aanmeldingsformulieren TAWN.
2. Thermische Analyse Nomenclature (van Ekeren)
3. DSC/TGA Cursus (herhaalde oproep)
4. Conferentie Data.
5. TAD 2009 (Noordwijk)
6. Reisbeurs TAWN
7. Bijdrage Firma's (Netzsch)
8. DMA of Composites (J. Schawe)
9. Websites.

THERMISCHE ANALYSE WERKGROEP NEDERLAND

Sinds 1965 bestaat er in Nederland een werkgroep (vanaf 1990 een officiële vereniging) genaamd Thermische Analyse Werkgroep Nederland, afgekort TAWN. Deze werkgroep heeft thans bijna 300 leden, die zich vanuit zeer verschillende onderzoeksgebieden bedienen van thermische analyse (DTA, DSC, TG, TMA, DMA, etc.) en calorimetrische technieken. De TAWN is lid van de internationale organisatie op het gebied van thermische analyse en calorimetrie, de ICTAC (International Confederation for Thermal Analysis and Calorimetry).

Doel van de TAWN
Het doel van de werkgroep is het bevorderen en verspreiden van kennis en kunde op het gebied van thermische analyse en calorimetrie. Om dit doel te bereiken worden er activiteiten georganiseerd, waar de leden onderling informatie kunnen uitwisselen met betrekking tot de mogelijkheden van thermische analyse en calorimetrie bij fundamenteel en toegepast onderzoek alsmede bij kwaliteitscontrole van producten.

Activiteiten
Jaarlijks wordt een thermische analysedag (TAD) georganiseerd. Daarnaast zijn er thema(mid)dagen over speciale onderwerpen. Tijdens deze bijeenkomsten houden leden of uitgenodigde sprekers voordrachten over hun werk. De toegang is voor leden gratis. Deze bijeenkomsten bieden uitstekende mogelijkheden om contacten op te bouwen met andere onderzoekers in hetzelfde vakgebied.
Daarnaast werkt de TAWN intensief mee aan cursussen op het gebied van de thermische analyse en calorimetrie.
Een aantal maal per jaar geeft de werkgroep een blad uit, het Thermische Analyse Bulletin. Dit blad wordt gratis naar de leden gestuurd.

LIDMAATSCHAP
Het lidmaatschap van de TAWN is slechts mogelijk voor natuurlijke personen; de contributie bedraagt
€ 10,-- per jaar. Opgave is mogelijk door de ingevulde aanmeldingsstrook te zenden naar de secretaris van de vereniging.
Sponsoring
Voor bedrijven en instellingen bestaat de mogelijkheid de werkgroep te sponsoren. Ook kunnen advertenties worden geplaatst in het TA-bulletin. Informatie hierover is verkrijgbaar bij de secretaris van de werkgroep of de redacteur van het TA-bulletin.

Aanmelding als lid van de TAWN

Ondergetekende geeft zich op als lid van de TAWN.

Naam: _____Hr./Mw. Titel(s):_____ Voorletters: _____

Bedrijf/Instelling:
_____

Afdeling:
_____

Adres: _____

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Telefoon: _____ Fax: _____ E-mail: _____

Handtekening:

Deze strook sturen naar de secretaris van de TAWN:

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ICTAC Nomenclature of Thermal Analysis
Dr. Paul van Ekeren (August 2006)

Lange tijd is er binnen de International Confederation of Thermal Analysis and Calorimetry (ICTAC), de internationale vereniging waarbij ook de TAWN is aangesloten, gesproken over een aanpassing van de nomenclatuur aan de meest recente ontwikkelingen, waaronder gemoduleerde temperatuur technieken en "sample-controlled techniques". In het bijzonder de nomenclatuurcommissie onder het voorzitterschap van prof. Hemminger, waarin ook ons bestuurslid Gerrit Hakvoort zitting had, heeft zeer uitgebreide voorstellen gedaan, die echter uiteindelijk de noodzakelijke goedkeuring niet hebben verkregen.
Een minder vergaand voorstel is in de council van de ICTAC besproken tijdens het ESTAC congres in Krakow (aug. 2006). Omdat er vooral door Duitsland en Nederland nog een paar bezwaren werden ingebracht waren er meer sessies noodzakelijk, maar gelukkig kon er toch overeenstemming worden bereikt. De definitieve tekst van de (nieuwe) ICTAC nomenclatuur vindt u hierna afgedrukt. De TAWN adviseert zijn leden om zoveel mogelijk van deze nieuwe nomenclatuur gebruik te maken.

Foreword

Since the formation of ICTA in 1968, continuous efforts have been made to standardize and improve the nomenclature used, because “it enables all scientists to speak the same language, it ensures there is only one term for each entity and it prevents different interpretations” (Mackenzie, 1969). Painstaking and laborious discussions by successive Nomenclature Committees have selected the most logical and acceptable terms and published them widely, particularly in “For Better Thermal Analysis” (1977), “For Better Thermal Analysis II” (1981) and “For Better thermal Analysis and Calorimetry III” (1991).

With such a wealth of information and definition, the need for a further document requires justification.

Firstly, since the report of 1991, many new developments in instrumentation and processing have been introduced. These have broadened the fields to which thermal analysis and calorimetry may be applied, and have greatly improved the sensitivity, reliability and ease of use of the techniques. Therefore, it is very important to include and distinguish these new methods.

Secondly, some of the earlier definitions should be reconsidered. In some instances, techniques are now used only rarely, while others are employed much more often, or in a different mode. Sometimes, the various names used by different workers can now be combined into a simple, comprehensive term, which covers many aspects.

Thirdly, the work should include the consideration of calorimetry, added to the remit of the Confederation, which changed in 1992 to ICTAC- International Confederation for Thermal Analysis and Calorimetry. This aspect has proved to require much more work, perhaps by calorimetry specialists in close collaboration with IUPAC.

Introduction

This document acknowledges the debt to previous members of the ICTAC Nomenclature Committees, initiated in 1965 under the guidance of Robert Mackenzie and with the secretarial expertise of Cyril Keattch. Succeeding Chairmen, including John Sharp (1984-8), Ed Gimzewski (1988-1992) and Wolfgang Hemminger (1992-2001) with their Committee members continued the discussions and published their findings. The task of the current committee has been to rationalise the work of all proceeding committees and to deliver a document that covers current practice in thermal analysis that can be accepted internationally.

Thanks are due to recent members of the Nomenclature Committee for their contributions to the deliberations and to others for the advice received: Roger Blaine (2001-4); Don Burlett (2001-4); Edward Charsley (2001-4); Valter Fernandez (2001-4), P.C. Gravelle (1992-2001); B.O.Haglund (1992-2001); Peter Haines (1997-2004, Secretary 2003-4); Gerrit Hakvoort (1992-2001); Wolfgang Hemminger (Chairman, 1992-2001); Trevor Lever (Chairman, 2001-4); Marianne Odlyha (1991-2001); Takeo Osawa (2001-4); Duncan Price (2001-4, Secretary, 2001-3); Michael Reading (1991-7); Stefan Sarge (1992-2001, Secretary, 2000-1); Judit Simon (1992-2001); Fred Wilburn (Secretary, 1991-2000)

In considering all the matters of nomenclature, the current Committee has followed the advice of the late Robert Mackenzie in that:

- **terminology should be simple;**
- **abbreviations kept to a minimum;**
- **names based on particular instruments should be discouraged.**

1. Scope

The scope of this document is to provide scientists working in the field of thermal analysis with a consistent "definitions of terms" that are commonly used within the field to allow precise communication and understanding.

2. Intent

This document acknowledges that nomenclature develops – without regulated definition – as the field of thermal analysis develops. Some terms used by authors and scientists rapidly become accepted by the scientific community, even if the term is not consistent with past definitions, science or

grammatically correct. However, if such a term is widely used and understood, it is reported here.

3. Definition of the field of Thermal Analysis (TA)

Thermal Analysis (TA) is the study of the relationship between a sample property and its temperature as the sample is heated or cooled in a controlled manner.

4. Techniques

A technique exists for each property or physical quantity that is measured versus temperature – a summary of some of these are presented below.

Property or physical quantity	Technique	Abbreviation(s)	Notes
Heat	Calorimetry		
Temperature	Thermometry		May also be described as heating or cooling curves.
Temperature Difference	Differential Thermal Analysis	DTA	A technique where the temperature difference between a sample and a reference material is measured
Heat Flow Rate	Differential Scanning Calorimetry	DSC	A technique where the heat flow rate difference into a sample and a reference material is measured.
Mass	Thermogravimetry or Thermogravimetric Analysis	TG TGA	The abbreviation TG has been used, but should be avoided, so that it is not confused with T_g (glass transition temperature)

Property or physical quantity	Technique	Abbreviation(s)	Notes
Dimensional and Mechanical Properties	Dynamic Mechanical Analysis	DMA	Moduli (storage / loss) are determined
	Thermomechanical Analysis	TMA	Deformations are measured.
	Thermodilatometry	TD	Dimensions are measured
Electrical Properies	Dielectric Thermal Analysis	DEA	Dielectric Constant/ Dielectric Loss measured
	Thermally Stimulated Current	TSC	Current
Magnetic Properties	Thermomagnetometry		Often combined with TGA
Gas flow	Evolved Gas Analysis	EGA	The nature and/or amount of gas / vapour is determined.
	Emanation Thermal Analysis	ETA	Trapped radioactive gas within the sample is released and measured.
Pressure	Thermomanometry		Evolution of gas is detected by pressure change.
	Thermobarometry		Pressure exerted by a dense sample on the walls of a constant volume cell is studied.
Optical Properties	Thermoptometry		A family of techniques in which an optical characteristic or property of a sample is studied
	Thermoluminescence	TL	Emitted light measured
Acoustic Properties	Thermosonimetry or Thermoacoustimetry		Techniques where the sound emitted (sonimetry) or absorbed (acoustimetry) by the sample is studied
Structure	Thermodiffractometry		Techniques where the compositional or chemical nature of the sample are studied
	Thermospectrometry		

5. Terminology and Glossary

NOTE: For all the techniques listed here, the terminology defines the property that is measured, and each definition may be completed by adding "as a function of temperature". For example: dynamic mechanical analysis (DMA), n- a technique where moduli are determined as a function of temperature.

adiabatic, ***adj- indicating that the experiment is carried out so that no heat enters or leaves the system.***

atmosphere, ***n- the gaseous environment of the sample, which may be controlled by the instrumentation or generated by the sample*****.**

calorimetry, ***n- techniques where heat is measured.***

combined, ***adj- the application of two or more techniques to different samples at the same time. This can include thermal and non-thermal analytical techniques e.g. TGA-FTIR.***

controlled temperature program, ***n- the temperature history imposed on the sample during the course of the thermal analysis experiment.***

cooling curve*, n- the experimental result of measuring the temperature of the sample as a function of time during cooling. The technique is thermometry, and heating curves are obtained for temperature-time experiments during heating.*

derivative, ***adj- pertaining to the 1st derivative (mathematical) of any curve with respect to temperature or time.***

dielectric thermal analysis (DEA*), n- a technique where dielectric properties are measured.*

differential, ***adj- pertaining to a difference in measured or measurable quantities usually between a sample and a reference or standard material.***

differential scanning calorimetery (DSC), ***n- technique where the heat flow rate difference into a sample and a reference material is measured.***

differential thermal analysis (DTA), ***n- a technique where the temperature difference between a sample and a reference material is measured.***

dynamic, ***adj- a prefix indicating that a parameter changes continuously during the experiment. The opposite of static.***

dynamic mechanical analysis (DMA), ***n- a technique where moduli are determined.***

emanation thermal analysis (ETA), ***n- a special type of EGA where the emanation of previously trapped radioactive gas is measured*****.**

evolved gas analysis (EGA), ***n- a family of techniques where the nature and/or amount of gas or vapour evolved is determined. The term evolved gas detection***
(EGD) has also been used where the nature of gas is not determined.

gas flow, ***n- the passage of gas from one part of the system to another, either by sorption by the sample, evolution from it, or chemical reaction*****.**

high pressure, ***(HP), adj- a prefix applied to the technique name to indicate that the pressure of the experiment is above ambient.***

> **Note: As an example a TGA experiment carried out under elevated pressures would be High Pressure Thermogravimetric Analysis (HP-TGA).**

isobaric, ***adj- a prefix indicating the experiment is carried out at constant pressure.***

isothermal, ***adj- a prefix applied to a technique to indicated that the temperature is maintained constant throughout the experiment.***

micro-, ***adj- a prefix used to denote that the technique measures small quantities, either with respect to the amount of sample studied, or with respect to the change in the properties measured.***

> **Note: This prefix has been applied to many thermal methods, and the equipment associated with them, for example micro-balance, micro-reactor, micro-calorimeter and also to the technique itself: micro-thermal, microscopic and the property studied: micro-structural.**

> **Note: The opposite prefix, macro- is also occasionally used.**

modulated, ***adj- a prefix indicating that a parameter changes in a periodic manner during the experiment.***

modulated temperature, ***(MT), adj- a prefix applied to the technique name to indicate that a temperature modulation has been applied to the temperature program.***

> **Note 1: As an example a DSC experiment carried out with a modulated temperature program would be Modulated Temperature Differential Scanning Calorimetry (MT-DSC).**

> **Note 2: Other modulated techniques are possible, such as modulated force TMA, modulated rate SCTA etc..**

> **Note 3: The use of the prefix MT is preferred to TM.**

photo-, ***adj- prefix to indicate that the experiment involves the illumination of the sample or measures the amount of light emitted from a sample. Where possible the wavelength range of the light should be specified.***

sample-controlled, ***adj- prefix applied to the technique to indicate that a property of the sample is used either continuously to control the sample heating. With no prefix, it is assumed that the experiment is following a controlled-temperature program.***

> **Note: the generic term for all TA techniques making use of such a feed-back is Sample- Controlled TA (SCTA), whereas specific names will be of the form Sample-Controlled TGA (SC-TGA) etc..**

scan, ***n- a term used to describe the data produced from a thermal analysis experiment. More correct usage is a thermal analysis curve, or, for a specific technique thermogravimetric curve, etc..***

scanning, ***adj- a prefix indicating a specified experimental parameter, usually temperature, is changed in a controlled manner.***

simultaneous, ***adj- the measurement of two or more properties of a single sample at the same time.***

> **Note: A hyphen is used to separate the abbreviations of the techniques; for example, simultaneous measurement of mass and heat flow rate (thermogravimetric analysis and differential scanning calorimetry) would be TGA-DSC.**

static*, adj- indicating a constant parameter during the experiment. The opposite of dynamic.*

stepwise, ***adj- prefix indicating discrete, discontinuous changes in an experimental parameter, e.g. force, temperature etc..***

tan ***δ*****,** ***n- is the dimensionless ratio of energy lost to energy returned during one cycle of a periodic process. For example tan δ = E'' / E', in DMA.***

temperature-programmed desorption (TPD), ***n- EGA using an inert atmosphere or vacuum, in the absence of sample decomposition.***

temperature-programmed oxidation (TPO), ***n- Experiment using an oxidising atmosphere, usually oxygen. Oxidation is monitored by any appropriate technique (EGA, TGA, gas sorption, etc.).***

temperature-programmed reduction (TPR), ***n- Experiment using a reducing atmosphere, usually hydrogen. Reduction is monitored by any appropriate technique (EGA, TGA, gas sorption, etc.).***

thermal curve, ***n- any graph of any combination of property, time, temperature derived from a thermal analysis technique.***

> **Note: thermal curve is a lose abbreviation of the more correct term thermoanalytical curve.**

thermally stimulated current (TSC), ***n- a technique where the current from the relaxation of sample polarisation is measured.***

thermo-, ***adj- a prefix indicating the use of changing temperature during the experiment.***

thermoacoustimetry, ***n- a technique where the characteristics of sound waves passing through the sample are measured*.**

thermoanalytical*, adj- of, or pertaining to, thermal analysis***.**

thermodiffractometry, ***n- a technique where the X-ray diffraction of the sample is measured.***

thermodilatometry,(*TD), n- a technique where one or more dimensions of the sample is measured under negligible load*

thermogravimetric analysis, ***(TGA), n- a technique where the mass of the sample is measured.***

thermogravimetry, ***(TG), n- see thermogravimetric analysis.***

thermoluminescence, ***n- a technique where light emission from the sample is measured.***

thermomagnetometry, ***n- a technique where a magnetic property of the sample is measured.***

thermomanometry, ***n- a technique where the pressure is measured.***

thermomechanical analysis, ***(TMA), n- a technique where the deformation of the sample is measured under constant load.***

thermometry, ***n- a technique where the temperature of the sample is measured.***

thermomicroscopy, ***n- a technique where the optical properties of the sample are observed and measured through a microscope.***

thermoptometry*, n- a technique where the optical properties of a sample are measured.*

thermosonimetry, ***n- a technique where the sound emitted by the sample is measured.***

thermospectrometry, *n- a group of techniques where a spectrum of the sample is measured.*

torsional braid analysis (TBA), *n- a dynamic mechanical analysis technique where the sample is supported on a braid.*

6. Experimental Conditions

The specifics of how the technique is used, additional experimental parameters and constraints should, of course, be reported alongside the data in all published work. It is important to separate the technique (instrumentation) from the way in which it is used (experiment). Of course, the make and model number should be included in all reports, papers and studies as well as an experimental section that describes in full all experimental parameters.

> **Note: For example in a Thermomechanical Analysis (TMA) experiment the sample may be subjected to no force, a constant force, an increasing force or a modulated force – or any combination of the above – during a single experiment. The technique (TMA) has not changed, only the experimental variables for that technique.**

The reader is referred to the ICTA publication "Reporting Experimental Results" (G. Lombardi 1977) for specific guidelines.

It must also be stressed that it should be normal practice to use the standard IUPAC quantities, units and symbols when reporting any work in thermal analysis. These are listed in: I.Mills, T. Cvitaš, K. Homann, N. Kallay and K. Kuchitsu (Editors), *Quantities, Units and Symbols in Physical Chemistry,* 2nd Edn., Blackwell Science/IUPAC, 1993 and other texts.

7. Symbols used specifically in Thermal Analysis.

The recommended SI symbols should be used.

Quantity	Symbol	Units
length	l	m
mass	m	kg, g, mg (etc).
time	t	s, (min, h)
current	I	A
temperature	T	K, °C
heating rate	$\beta = (\mathrm{d}T/\mathrm{d}t)$	$K\ s^{-1}$
fraction reacted	α	-
heat	q, Q	J
heat flow rate	$\Phi = (\mathrm{d}q/\mathrm{d}t)$	W
heat capacity at const. pressure	C_p	$J\ K^{-1}$
Quantity	**Symbol**	**Units**
heat capacity at const. volume	C_V	$J\ K^{-1}$
pressure	p	Pa
modulus of elasticity	E	Pa

Symbols describing specific events or materials.

- **In general, symbols for physical quantities should be in *italic* type, or, if vectors, in *bold italic type.***
- **Units do not take plural.**
- **Subscripts should be restricted to single letters.**
- **If the subscript relates to an object or property, it should be a CAPITAL letter:**
  - m_S = mass of sample S.
  - T_R = temperature of reference R.
  - T_C = Curie temperature
- **If the subscript refers to a phenomenon, it should be lower case:**
  - T_m = melting temperature
  - T_g = glass transition temperature.
- **If the subscript refers to a specific event, time or point, it should be lower case or figures:**
  - T_i = initial temperature
  - m_f = final mass
  - T_p = peak temperature
  - $t_{½}$ = time of half reaction
- **Changes in extensive thermodynamic quantities X due to an event y should be represented by $\Delta_y X$:**
  - $\Delta_{vap}H$ = enthalpy of vaporization
  - $\Delta_r G$ = Gibbs energy of reaction.

- **Symbols for the physical state of the material should be put in brackets after the formula symbol:**
  - $\Delta_{vap}H = H(g) - H(l)$

8. Overview

This document is concerned with providing definitions of common terms that are used by thermal analysts to report, present and explain their work.

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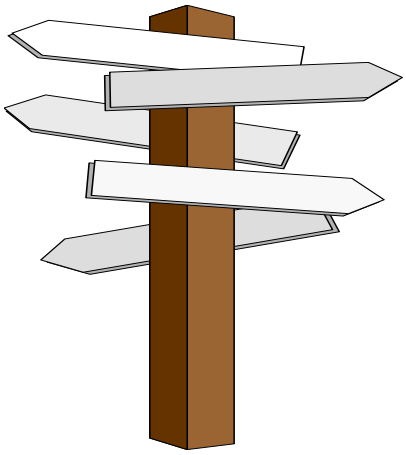
U KUNT NOG STEEDS AAN HET BESTUUR DE BEHOEFTE KENBAAR MAKEN VOOR EEN THERMISCHE ANALYSE CURCUS: DSC OF TGA.

CONGRESSEN, SYMPOSIA, CURSUSSEN



NATAS 37th Annual Conference - Exploring the Frontiers of Thermal Analysis and Rheology
Lubbock, USA, 21.09. - 23.09.2009
http://www.natasinfo.org/Natas2009(web)1.html
sindee.simon@ttu.edu

Thermosets 2009
Berlin, 30.09. - 02.10.2009

http://www.thermosets.de/index.html
info@thermosets.de

GEFTA Jahrestagung 2009
Gießen, D, 07.10. - 09.10.2009
http://www.gefta.org
gefta2009@arbmed.med.uni-giessen.de

PhandTA 11 - 11th Conference on Pharmacy & Applied Physical Chemistry
Innsbruck, CH, 07.02.2010 - 10.02.2010
http://www.eurostar-science.org
erwin.marti@apch.ch

7th Heat Flow Calorimetry Symposium
Rijswijk, NL, 17.05 - 20.05.2010

wim.deklerk@tno.nl

13th International Symposium on Loss Prevention and Safety Promotion in the Process Industries
Brugge, B, 06.06. - 09.06.2010
https://www.ti.kviv.be/conf/Lossprevention2010/
info@lossprevention2010.com

ESTAC-10 (10th European Symposium on Thermal Analysis and Calorimetry)
Rotterdam, NL, 22.08. - 27.08.2010

De Klerk / van Ekeren (TNO) wim.deklerk@tno.nl

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TAD 2009

DE TAD 2009 ZAL PLAATS VINDEN OP DINSDAG 24 NOVEMBER BIJ ESTEC IN NOORDWIJK. GRAAG ZO SPOEDIG MOGELIJK UW BIJDRAGEN KENBAAR MAKEN AAN HET BESTUUR.

HET PROGRAMMA EN NADERE DETAILS ZULLEN DAN IN HET VOLGENDE BULLETIN WORDEN GEPUBLICEERD EN OP DE WEBSITE VAN DE TAWN.

Reglement Reisbeurzen voor Jonge Onderzoekers

1. **Om het oordeelkundig gebruik van Thermische Analyse en/of Calorimetrie in Nederland en Vlaanderen te stimuleren kan het bestuur van de TAWN een beurs toekennen aan jonge onderzoekers die op een (inter)nationaal congres of symposium resultaten van hun werk willen presenteren door middel van een lezing of een poster.**
2. **Het bestuur van de TAWN zal zich inzetten om deze mogelijkheid bekend te maken.**
3. **Het te presenteren werk moet voor een significant deel bestaan uit het correct toepassen van Thermische Analyse, zoals DSC, DTA, TG, TMA, DMA en DEA, of calorimetrie.**
4. **Het budget dat voor de beurzen beschikbaar is wordt door het bestuur van de TAWN vastgesteld. Het bestuur zal trachten om het budget via één of meer sponsors bijeen te brengen.**
5. **De frequentie van toekenning van beurzen is budgetgebonden, en daardoor mede afhankelijk van de sponsormiddelen. In principe wordt gestreefd naar een jaarlijkse toekenning van een beurs.**
6. **Een beurs kan op ieder gewenst tijdstip worden toegekend aan een kandidaat die een aanvraag indient bij het bestuur. De aanvraag dient te zijn voorzien van een 'abstract' van de presentatie, aangevuld met relevante informatie om de aanvraag te kunnen beoordelen. Tevens dient een begroting van de kosten te worden overlegd.**
7. **De kandidaat voor een beurs dient de leeftijd van 35 jaar nog niet te hebben bereikt.**
8. **Over toekenning van een beurs wordt beslist door het bestuur van de TAWN op basis van de kwaliteit van de voorgestelde presentatie.**
9. **Wanneer een bestuurslid zelf is betrokken bij het werk van een kandidaat, dan zal hij wel mee kunnen overleggen en adviseren, maar niet deelnemen aan een eventuele stemming over toekenning van een beurs.**
10. **De beurs bestaat uit een geldbedrag. De hoogte van dit bedrag wordt bepaald door het bestuur, maar zal nimmer meer bedragen dan het totaal van de inschrijvings-, reis- en verblijfskosten.**
11. **In geval van sponsoring worden de sponsors vermeld bij de uitreiking van de beurs en bij de presentatie.**
12. **Van degene die een beurs krijgt toegewezen wordt verwacht dat hij/zij een voordracht zal houden over zijn/haar werk tijdens een door de TAWN georganiseerde Thermische Analyse bijeenkomst in Nederland of Vlaanderen.**
13. **Een persoon kan slechts eenmalig een beurs ontvangen.**

14. **Het TAWN-bestuur is niet gebonden opening van zaken te geven over de besluitvorming.**
15. **In gevallen waarin dit reglement niet voorziet beslist het TAWN-bestuur.**

Vastgesteld te Utrecht,
in de TAWN-bestuursvergadering op 21 september 2006.

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Bijdragen TA firma's (buiten verantwoordelijkheid van redactie)

technex bv

NETZSCH Analyzing & Testing Acquires World's Most Advanced Adiabatic Reaction Calorimeters from TIAX

The NETZSCH Analyzing & Testing Business Unit recently announced its acquisition of the TIAX Accelerating Rate Calorimeters and Automatic Pressure Tracking Adiabatic Calorimeters (APTAC).
These products will be merged with NETZSCH's worldwide Thermal Analysis business.

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Adiabatic reaction calorimeters help secure safe and profitable operation in industry. As highly versatile, miniature chemical reactors, they measure thermal and pressure properties of exothermic chemical reactions. The resulting information helps engineers and scientists identify potential hazards and address key elements of process safety design including emergency relief systems, effluent handling, process optimization, and thermal stability. Industries such as the chemicals, pharmaceuticals, and energy branches as well as government agencies and laboratories use adiabatic reaction calorimeters to study chemical kinetics, storage and transportation issues, process interruptions, and chemical process design. Adiabatic reaction calorimeters are also used to investigate accidents and develop air bags, rechargeable batteries, spacecraft and rocket propulsion.

About the NETZSCH Analyzing & Testing Business Unit:
The NETZSCH Analyzing & Testing Business Unit offers the broadest range of products and testing services for Thermal Analysis and Thermophysical Characterization available through its extensive worldwide sales and service network. It was created in 1962 and has established itself as one of the world's best-known suppliers of high-performance thermoanalytical instruments.

About TIAX LLC:
TIAX LLC is a technology processing consulting company operating at the intersection of business and technology. The company transforms emerging innovations into robust technology platforms ready for hand-off and has more than 50 research and development laboratories.

Adiabatic Reaction Calorimetry

Accelerating Rate Calorimeters help industry operate safely and profitably. As highly versatile, miniature chemical reactors, they measure thermal and pressure properties of exothermic chemical reactions. The resulting information helps engineers and scientists identify potential hazards and address key elements of process safety design including emergency relief systems, effluent handling, process optimization, and thermal stability.
The patented VariPhi™ option enables for all Accelerating Rate Calorimeter / Automatic Pressure Tracking Adiabatic Calorimeter systems DSC like performance in the scanning mode: exotherms, endotherms and heat capacity plus pressure data.

Accelerating Rate Calorimeter 244

The cost-effective Accelerating Rate Calorimeter 244 is designed to safely measure the amount and rate of heat release associated with the processing or storage of chemicals with a sample volume of 0.5 ml to 7 ml. The key features are high performance, safety, usability, and flexibility with data integrity and robustness in a temperature range from ambient to 500°C.

Accelerating Rate Calorimeter 254

This new Accelerating Rate Calorimeter (model 254), formerly known as TIAX New ARC 5000, is a specialized instrument to help industry operate safely and profitably. Highly versatile, miniature chemical reactor, it measures the thermal and pressure properties of exothermic chemical reactions. The resulting information helps engineers and scientists identify potential hazards and tackle key elements of process optimization and thermal stability.

APTAC 264 - Automatic Pressure Tracking Adiabatic Calorimeter

APTAC 264 is capable of studying exothermic reactions at temperatures from ambient to 500ºC and pressures ranging from vacuum to 140 bar (2,000 psia). As a calorimeter, APTAC 264 can detect and track exotherms at heat generation rates ranging from 0.04 K/min to 400 K/min.

For more information, please contact Technex, Loran Mak, tel 075 647 4567, info@technex.nl

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Informatie over producten , adressen en telefoonnummers van bovenstaande firma's vindt U op de betreffende websites verder in dit Bulletin.

DMA of Composites: Selection of the Optimal Measurement Mode

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ABSTRACT

Dynamic mechanical analysis, DMA, is used to determine the glass transition temperature and the mechanical quantities of filled or fiber reinforced polymers, for example. These results are used to characterize the quality and stability of the composites, including bonds and lacquers. The characteristic glass transition temperature, T_g, is assigned by various standard methods, for example using an onset on the storage modulus, or the maximum temperature of tan δ or the loss modulus.

Using reactive processes like curing, it will be shown how DMA can deliver accurate information about the stability of materials, if an appropriate measurement mode is applied. Even sandwich systems (polymers with metals or fabrics), adhesives in bonded structures, lacquers and coatings can be optimally analyzed by DMA. The influences of the different deformation modes, bending and shear, are discussed on the basis of physical principles.

INTRODUCTION

Composite materials (or composites for short) are engineered materials made from two or more constituent materials with significantly different physical or chemical properties and which remain separate and distinct on a macroscopic level within the finished structure. One of the advantages of composites is that two or more materials could be combined to take advantage of the good characteristics of each of the materials. Usually, composite materials will consist of two separate components, the matrix and the filler. The matrix is the component that holds the filler together to form the bulk of the material. It usually consists of various polymers like thermosets, thermoplastics or elastomers, but other materials may be used. The filler is the material that has been impregnated in the matrix to lend its advantage (usually strength) to the composite. The fillers can be of any material such as carbon fiber, carbon black, glass bead, or sand. Composites can be classified into roughly three types according to the filler types: Particulate, short or long fiber and laminate. Here, we consider coatings in a similar manner, i.e. as a combination of a carrier matrix and cover layer. The main arrangements are:

- **Filler reinforced composites**
- **Fiber reinforced composites**
- **Laminates**
- **Coatings**

The reinforcement is usually much stronger and stiffer than the matrix, and gives the composite its good properties. The matrix holds the

reinforcements in an orderly pattern. Because the reinforcements are usually discontinuous, the matrix also helps to transfer load among the reinforcements.

Due to the high content of inert filler, the glass transition can often not be determined accurately enough using DSC or TMA. Hence, dynamic mechanical analysis, DMA, is used to determine the glass transition temperature and the mechanical quantities of the composites. These results are used to characterize the quality and stability of the composites. The characteristic glass transition temperature is assigned by various standard methods, for example using the maximum temperature of tan δ. For successful DMA measurements, it is important to choose the "right" experimental parameters. In practice, the difficulty is that the deformation mode, sample geometry and mechanical measurement parameters (force and displacement amplitude) are interdependent. This is even more important when measuring composites which may be isotropic or anisotropic in filler orientation. Depending on the sample geometry, orientation and deformation mode the anisotropic composites will give different results even for the matrix properties.

EXPERIMENTAL

The measurements were carried out using the METTLER TOLEDO STAR^e system and the DMA/SDTA861^e measuring cell with accessories for shear, bending and tension measurement.

The materials used and the measurement conditions are specified in the respective sections.

RESULTS AND DISCUSSION

Orientation of filler

Isotropic materials have properties which are invariant with respect to direction. Composites are filled with powders or short fibers with random distribution. In this case, the mechanical modulus measured by shear and tension are related by the Poisson's ratio, μ, which describes the volume change and, hence the relation between shear modulus, G, and Young's modulus, E (equation 1). In case of incompressible materials μ=0.5 and E=3G. Figure 1 shows this relationship if elastomers with nano-filler are measured.

$$G = \frac{E}{2(1+\mu)} \qquad (1)$$

With anisotropic materials the properties depend on the direction of the fillers, for example of fiber reinforced composites, laminates and coated films. The Poisson's ratio can get rather large so that Young's modulus can be much bigger than the shear modulus, E>(3G…..100G). A typical material for this behavior is a fiber reinforced epoxy matrix. As shown in Figure 2 the factor of 5.5 holds for the temperature range when the matrix is in the glassy state. But after glass transition the factor is not any more constant.

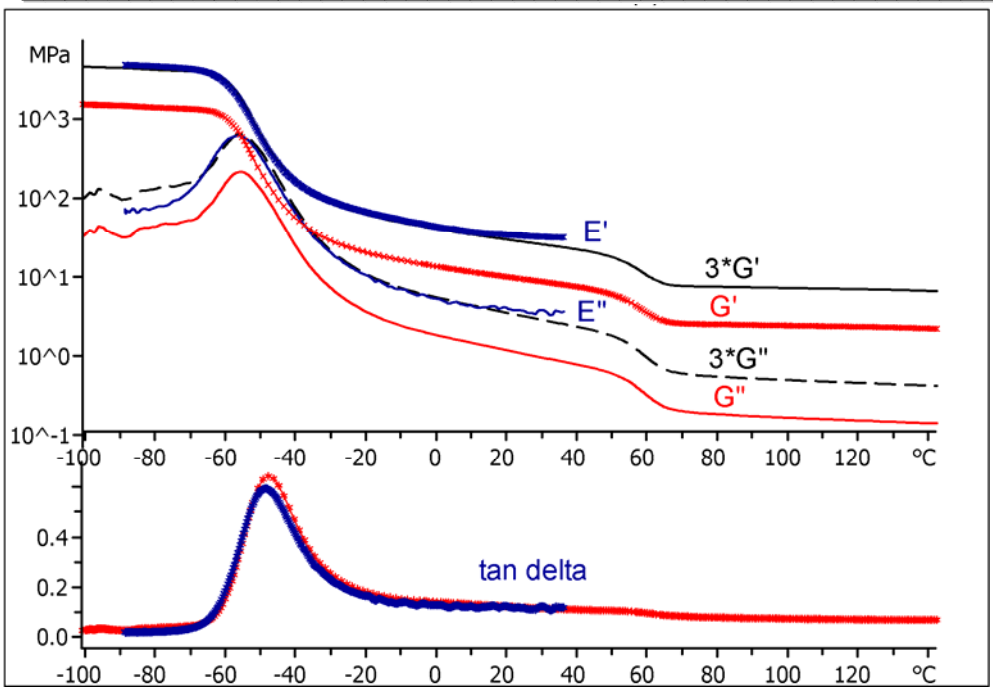

Figure 1. Isotropic composite: an elastomer filled with nano-particles is measured in tensile mode (E) and shear mode (G). The black lines show the shear modulus G multiplied by three. These curves fit the tensile modulus E in the range from -90 to 10 °C.

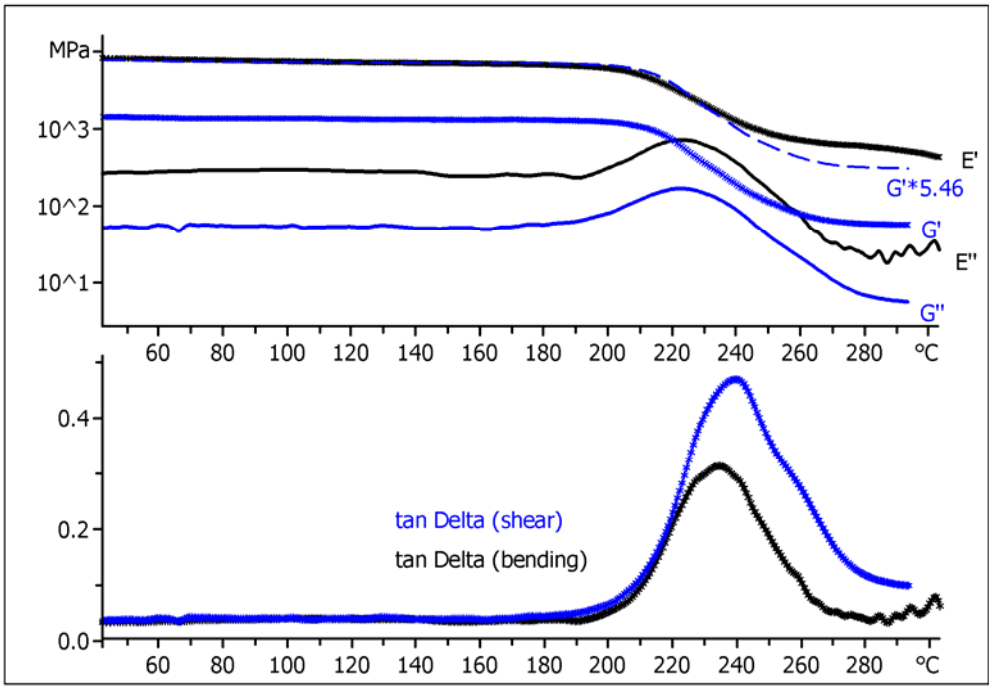

Figure 2. Anisotropic composite: fiber-reinforced epoxy is measured in tensile and shear mode. The dashed curve shows the shear storage modulus multiplied by the factor of 5.46 to fit the Young's storage modulus *E'* below glass transition.

Modulus and compliance

The mechanical behavior can be discussed either on the basis of elasticity with the description of the Young's modulus or shear modulus, for example as complex values *E and *G** respectively. Or, the compliance of a material can be investigated, for example the complex shear compliance *J** or the respective compliance for tension, compression or bending *D**. Modulus and compliance are equivalent as described by the following equations:**

$$J^*(\omega)=\frac{1}{G^*(\omega)} \quad \textbf{(2)}$$

$$D^*(\omega)=\frac{1}{E^*(\omega)} \quad \textbf{(3)}$$

$$J'=\frac{G'}{G'^2+G''^2} \quad \text{and} \quad J''=\frac{G''}{G'^2+G''^2} \quad \textbf{(4,5)}$$

$$J^*(\omega)=J'(\omega)-\mathrm{i}\,J''(\omega) \quad \textbf{(6)}$$

The equivalence is shown by the DMA curves in Figure 3. It has to be noticed that characteristic temperatures depend very much on the respective curve, for example the maximum of *G*" is at about 0 °C (at 1 Hz) but the maximum of *J*" is at 25 °C. Therefore it is important to compare results only if the materials have been measured under the same conditions and if the same curves are discussed.

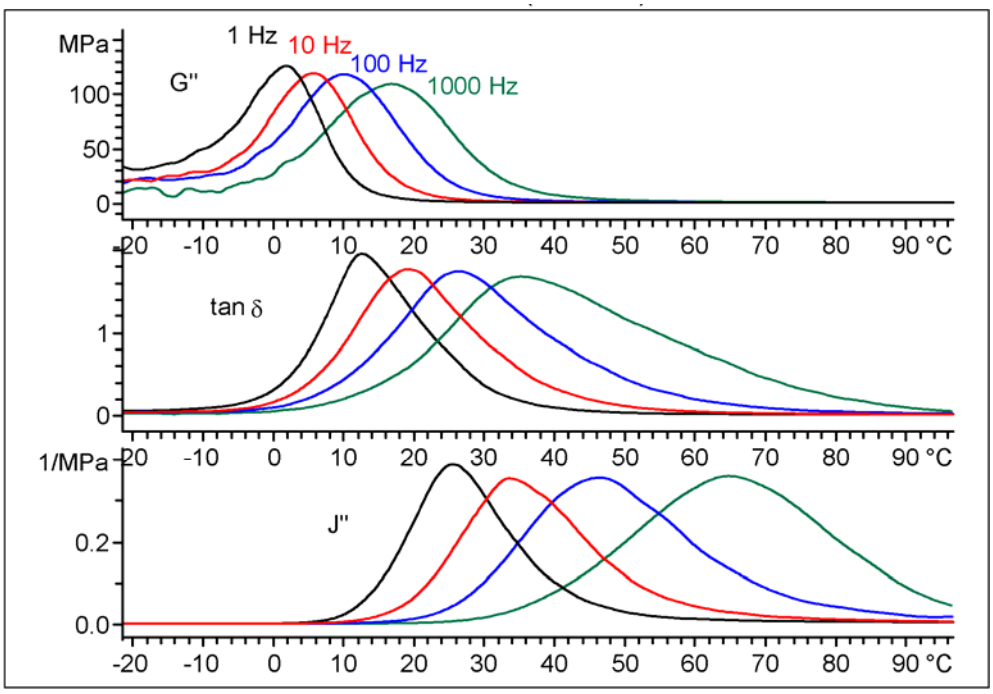

Figure 3. Modulus and compliance: DMA curves of a vulcanized SBR elastomer measured in shear mode at different frequencies. Different peak temperatures can be reported depending on the curve evaluated.

The influence of orientation has an important impact on the detection of effects and on the selection of the optimal measurement mode and display of measurement curve. This is demonstrated by a simple example of an anisotropic material having just two components with different mechanical parameters as it is the case for coatings or laminates, see Figure 4.

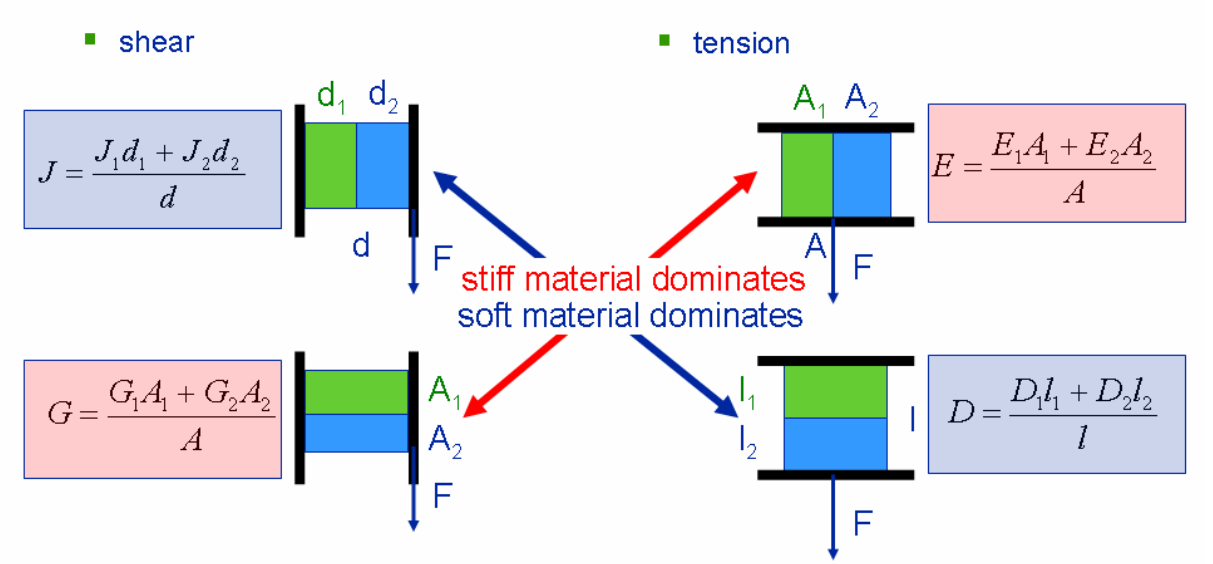

Figure 4. Example of a simple composite to show in which arrangement the properties, modulus or compliance are additive.

An example of such a system is a composite consisting one very stiff part; like a metal sheet or carbon fibers. The other is a matrix resin which softens during glass transition. If the matrix has to be checked for it's curing property only the arrangement shown in the picture on top left or bottom right can be used to clearly show the changes, for example to identify a glass transition temperature. As an example Figure 5 shows the compliance with two peaks representing the glass transitions of the two rubbers in the composite of metal/rubber/textile/rubber laminate.

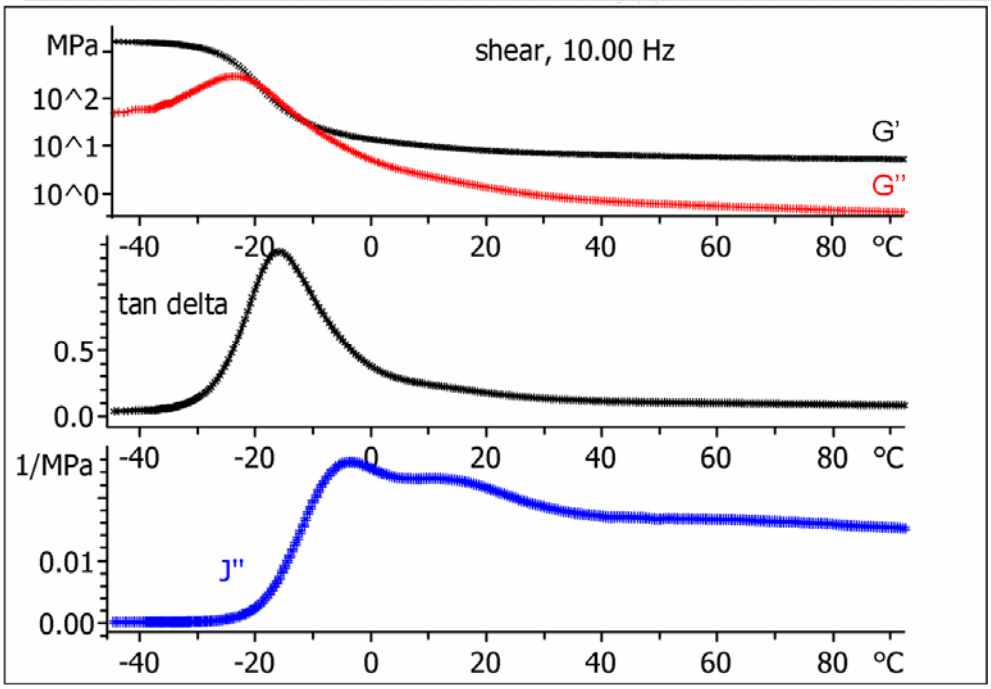

Figure 5. Example of a metal/rubber/textile/rubber composite measured in shear mode. The compliance *J"* shows two peaks which are not shown by *G"* or tan δ.

Young's modulus by bending

Bending measurements are much more complex to be described. Nevertheless, bending modulus is measured often in practice to characterize the property of the composite and changes due to temperature change. But again, the anisotropic behavior has to be considered for the determination of the Young's modulus as well as for the glass transition temperature. With very long fibers and ideal interaction between fiber and matrix the modulus in fiber direction can be described by a weighed sum of the modulus of the fibers, E_f, and the modulus of the matrix, E_m, with f as the filler content:

$$E = E_m(1-f) + E_f f \qquad \textbf{(7)}$$

E_f is usually much bigger than E_m and E is therefore close to E_f.

If bending measurement is done with the force across the fiber direction, the resulting modulus is close to the modulus of the matrix:

$$E \approx E_m/(1-f) \qquad \textbf{(8)}$$

This behavior is shown in Figure 6 using a polyethersulfone (PES) with reinforcement fibers. The change in modulus during the glass transition is across fiber direction much bigger than in the other direction. But this influences also the peak temperature of tan δ. Even if the shift of 6 K is not much it could decide about good or bad quality of the composite. The peak temperature of *E"* is not affected.

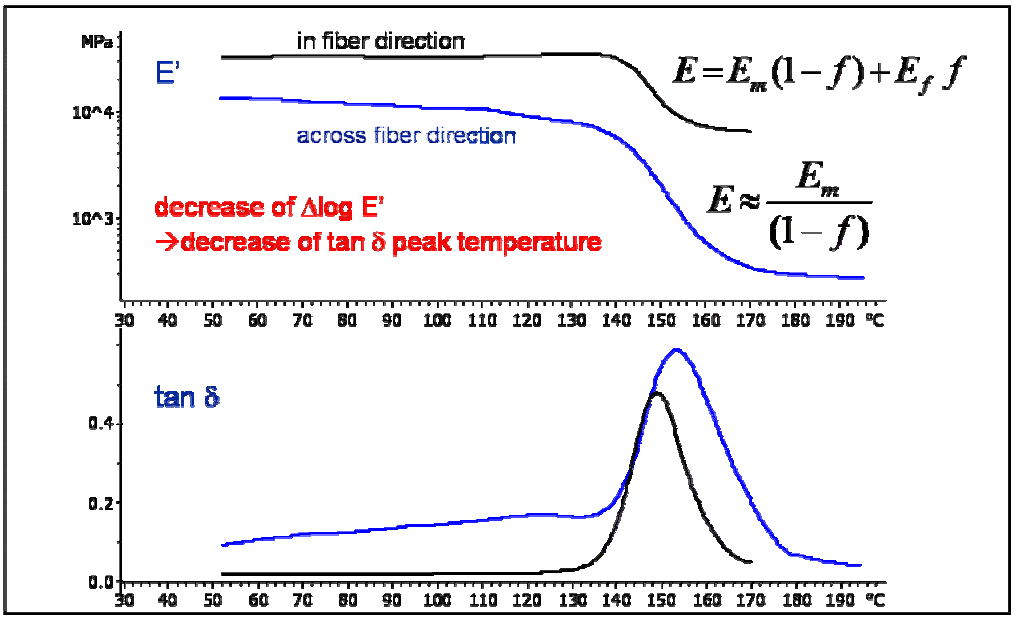

Figure 6. DMA of a PES composite with fiber reinforcement. Bending measurements in and across fiber direction. The shift of the peak of tan δ is about 6 K.

Coatings and adhesives

Coatings and adhesives are usually thin layers on a thick carrier film or sheet (matrix). Bending measurement is often not sensitive enough to determine the properties of the coating on a stiff matrix. For tensile measurements the coating should be removed from the matrix what is usually very difficult. Due to the thin coating, compression measurements are also not useful. Therefore, shear deformation is best suited to analyze thin coatings on a stiff matrix. An example is given in figure 7.

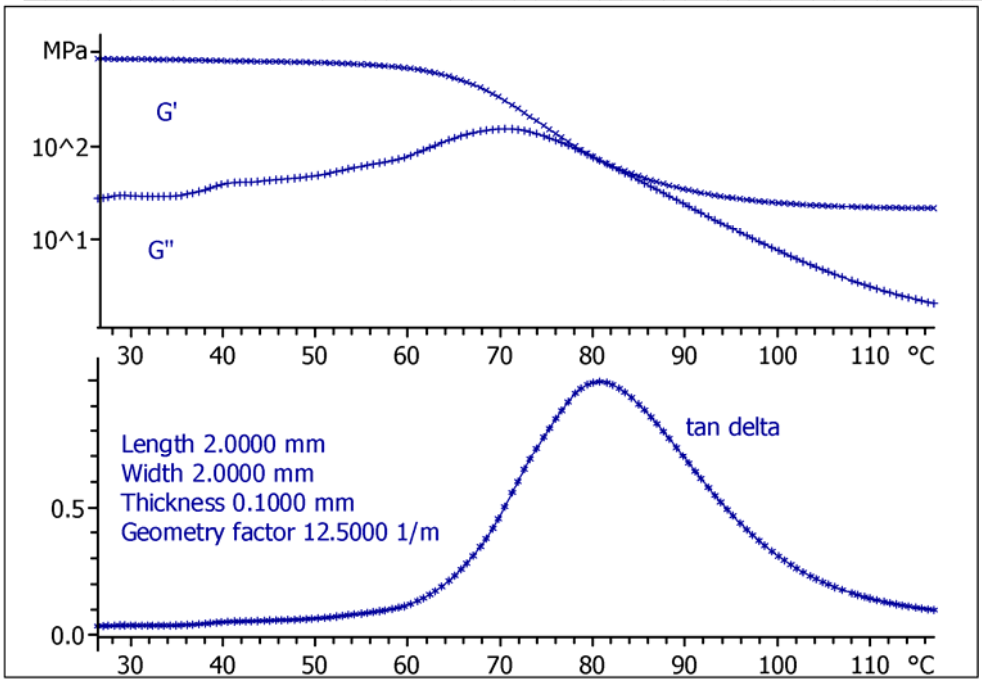

Figure 7. DMA of a coating on a metal sheet using the shear mode. The sample geometry has to be optimized together with the modulation conditions. The thickness of 0.1 mm is the thickness of the coating only.

CONCLUSIONS

DMA measurements are used to characterize the mechanical behavior, the quality and the stability of the composites, including bonds and lacquers. Due to the sample geometries bending, tensile or shear deformation is usually applied. To get optimal results it has to be considered that:

- **Composites are inhomogeneous materials**
- **Composites shows often non-linear behavior**
- **Composites are often anisotropic.**

Typical problems are related to reinforcement, glass transition, stability of the matrix and the interaction between matrix and filler. The deformation mode and the sample stiffness (geometry) has therefore to be selected carefully and has to be adequate for the measurement task:

- **3-Point bending: for the measurement of the reinforcement of fibers and the modulus of bulky material having high stiffness. But the peak temperature of tan δ depends on the modulus change and the modulus is influenced by the geometry change.**
- **Tension: for Young's modulus of thin films**
- **Shear: for determination of the characteristic glass transition temperature of the matrix, for laminates, "sandwich systems" and adhesives. Using small specimen from different parts of a samples the homogeneity of the composite can be checked. High temperature accuracy is reached by direct contact of the temperature sensor with the clamps holding the samples.**
  The peak temperature of tan δ depends mainly on the degree of curing of the matrix and not on the modulus change.

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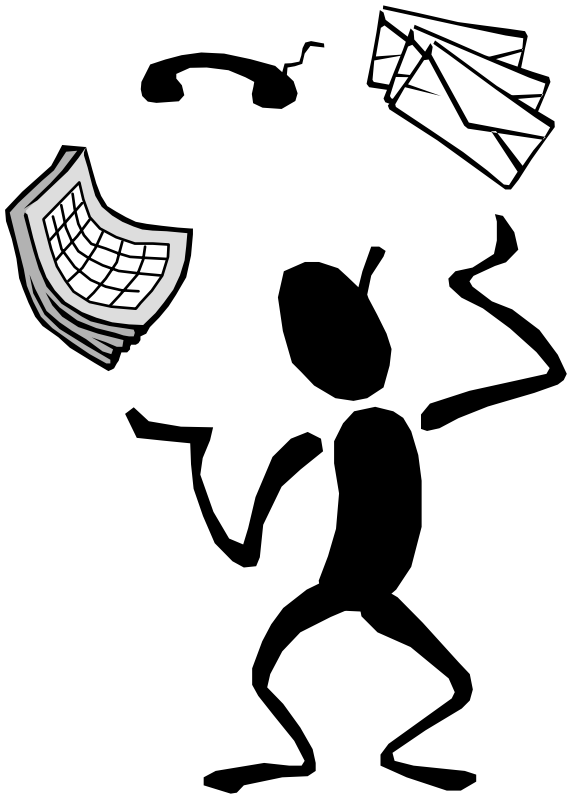

Thermische Analyse Bulletin

Jaargang 31