Functional measurement in nanomaterials using optical and thermal techniques.

Various authors edited by Patrick Courtney

For further information please contact Dr Patrick Courtney at Patrick.courtney@perkinelmer.com Tel +44 1494 679078

1 Introduction

Advanced instrumentation is key to work in nano-materials. Functional tools such as optical and thermal measurement techniques allow the characterisation of materials. As such, they complement imaging tools such AFM and TEM-SEM which give spatial informätion on the structure of the materials.

This poster presents examples of recent challenging measurements carried out in band gap analysis, plasmon resonance and polymorphic structure.

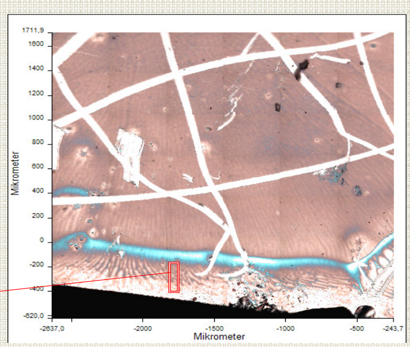
2 Collaborative development

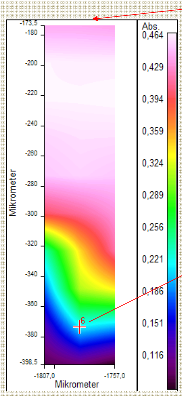
In some instances, no suitable measurement method or instrumentation exists. In this case it is necessary to adapt an existing method or develop an entirely new one.

In order to address such situations, Perkinelmer is open to working collaboratively in areas of joint interest. A dedicated applications laboratory is available, providing state-of-the art hardware and software capability for development work as well as access to multi-disciplinary applications scientists, assisting with the more advanced techniques, to get the best results from novel or difficult samples. We have an extensive network of like-minded collaborators, who can offer support & guidance.

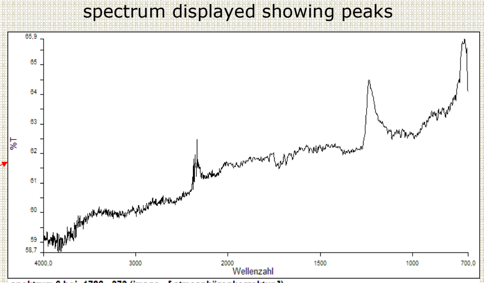
3 Studies of nanomaterials using FTIR imaging

Prof. Taubner wants to develop novel materials exhibiting meta-material properties. For this reason, he needs to perform IR spectroscopy of arrays of Au nanostructures embedded in a 50nm thin PMMA film. Examination with an FTIR imaging instrument shows characteristic peaks of PMMA absorption which might already be enhanced by plasmon resonance.





Region of sample selected for mapping and IR spectrum displayed showing peaks



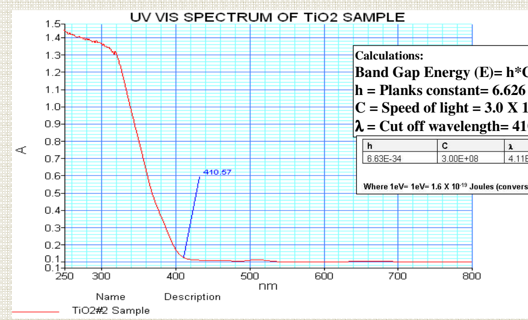
All data courtesy of Prof. Taubner University Aachen Germany.
Data collected by Bernd Dippel, Perkinelmer

4 Band gap measurement by Spectroscopy

One of the most common measurements made is the determination of Band Gap properties of a material. This relates to the ease in which a semiconducting material will transfer electrons to it's conducting band. This is fundamental to research on new materials ....

The solar spectral range, from 350-2500nm is used for the analysis of most materials. This includes not only the PV material but other components such as glass substrates and coatings. The Lambda 1050 equipped with a 150mm integrating sphere is one of the most common platforms to measure the diffuse reflectance and transmission characteristics of solar materials.

Titania (TiO2) is used here to summarize the typical process for determining the Band Gap of a material. The sample was measured in diffuse reflectance mode on a Lambda 1050 with the data recorded in absorbance. From the spectra below the wavelength at which the sample begins to absorb can clearly be determined. This wavelength is the Cutoff wavelength and is used in the equation below.



Calculations:
Band Gap Energy (E)= h*C/λ
h = Planks constant= 6.626 X 10-34Joules Sec
C = Speed of light = 3.0 X 10 8 meter /sec
λ = Cut off wavelength= 410.57 X 10-9 meters

h	C	λ	E	eV
6.63E-34	3.00E+08	4.11E-07	4.84156E-19	3.025976

Where 1eV= 1eV= 1.6 X 10^{-19} Joules (conversion factor)

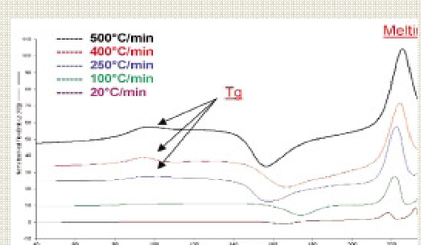
The band gap is the potential needed to move electron to the conduction band expressed in eV.

Data courtesy Chris Lynch, Frank Padera, Bill Sweet, Aniruddha Pisal of Perkinelmer

5 Nanostructure by determination Tg

One important characteristic of any nano-material is its nano-crystalline structure. One convenient way of accessing this is via measurement of the glass transition Tg using a technique such as differential scanning calorimetry (DSC).

There has been great interest over the years in the study of the Tg of amorphous lactose. Lactose is a very important excipient for pharmaceuticals and is used widely as a diluent in the formulation of tablets as well as being used as a carrier in dry powder inhalation (DPI) products. Because of its wide use as an excipient, information about the form in which it is manufactured is critical. Changes in its structural form can lead to changes in the characteristics and performance of a formulation, e.g., agglomeration within the powder of a DPI. This is an example of a case in which the re-crystallization from small domains of amorphous material can cause a major effect on the performance of a pharmaceutical. There are many examples in the literature where instability in the physical structure of the active pharmaceutical ingredient leads to changes in bioavailability.



The Tg of lactose is normally seen in the temperature range of 100–120 °C and is difficult to identify using conventional DSC scan rates.

However the increased sensitivity that comes from using HyperDSC allows the identification and quantification of the Tg event.

Data courtesy of Paul Gabbott, Paul Clarke, Tim Mann, Paul Royall & Sukhraj Shergill

PerkinElmer, Inc., 940 Winter Street, Waltham, MA USA (800) 762-4000 or (+1) 203 925-4602 www.perkinelmer.com