Thermal Analysis Unit

Introduction

The Thermal Analysis Unit (TAU) employs some common techniques to monitor changes in the physical properties of materials, induced by a controlled heating of cooling program or following interactions with one another (reaction, dissolution, adsorption etc.). The basic techniques used in the TAU, as well as the properties being measured, are listed in Table 1.

Table 1. The TAU techniques

Technique	Measured property
Thermogravimetry (TG)	Mass
Derivative Thermogravimetry (DTG)	Mass
Differential Thermal Analysis (DTA)	Temperature
Differential Scanning Calorimetry (DSC)	Enthalpy
Thermodilatometry (TD)	Dimensions
Dynamic Mechanical Analysis (DMA)	Deformation/ Viscoelasticity

Facilities & Infrastructure

The following equipment is avalaible at the TAU:

- The thermal analysis instrument STA 449C *Jupiter*® (Fig. 1), designed to simultaneously measure the mass changes (TG) and the calorimetric effects (DSC or DTA) at both high and low temperature. The TG technique measures the temperature-induced changes in sample mass. The output signal may be differentiated electronically to yield a DTG curve. The TG technique may be used to study the dehydration, decomposition and thermal stability of solids, to determine their oxidation behaviour and specify the production processes of ceramics and other synthetic materials. Additional information on kinetics and reaction mechanisms may be deduced by treating the TG curves with different methods.
- The DTA technique measures the difference in temperature between the sample and a reference material. DTA curves provide information on the temperature range wherein a process takes place and allow calculation of the value of the enthalpy change (ΔH). The DSC technique, although being similar to the DTA, is more accurate and further determines the heat capacity of the samples. Application of both techniques is very widespread and may be employed in biotechnology, pharmaceutics and foods, in the study of polymer, inorganic and metallic materials, phase diagrams and purity determination.
- The thermodilatometric instrument DIL 402C (Fig. 2) measures the expansion or shrinkage of a material, which is subject to a controlled temperature-time program. Dilatometric measurements are applicable in ceramics and glass studies, as well as in densification and sintering studies of powders. Both instruments are connected with a data acquisition and control unit, as well as with a PC through the SW/STA/652.01 software, which is used for the analysis of results. Additional software packages are available, namely PEAKSEP and KINETICS, which perform peak separation of curves and kinetics analyses, respectively.

Figure 1: The STA 449C JUPITER equipment that combines TG/DTG, DTA and DSC techniques (on the right) and the SETARAM C80 calorimeter (on the left).

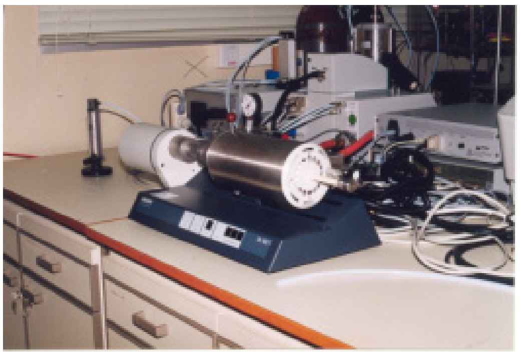

Figure 2: Thermodilatometry equipment, NETZSCH model DIL 402 C.

- The C 80 II calorimeter (Fig. 1) is used to measure the heat exchange and may provide useful information on the behaviour and thermal properties of the materials (such as fusion, polymerization, decomposition, oxidation, heat capacity) or the materials drought together (reaction, dilution, hydration, wetting). The instrument is also suitable for simulating mixing reactions (liquid-liquid, liquid-solid) and thermal transformation.

The above equipment is connected to a data acquisition and control unit, as well as to a PC equipped with the SW/STA/652.01 software, which may be used for data analysis and kinetics calculation.

- The DMA 242C (NETZSCH) instrument is used to measure the mechanical properties of materials, under an oscillating stress as a function of temperature, time and frequency (DIN 53513, DIN 53440, DIN-IEC 1006, ASTM D 4065, ASTM D 4092, ASTM D 4473, ASTM D 5023, ASTM D5024, ASTM D 5026, ASTM D 5418). Structural changes, such as the glass-transition temperature, secondary transitions, cross-linking etc, may thus be determined.

Services

The Unit is engaged in activities, such as:

University research activities

Department of Chemistry

- Laboratory of Industrial Chemistry (materials, catalysts, polymers and minerals)
- Laboratory of Inorganic Chemistry (complexes and zeolites)
- Laboratory of Physical Chemistry (materials)

Department of Physics

- Laboratory of Materials Science (superconductors, semi-conductors, polymers)

Department of Materials Science & Engineering

- Materials' studies, synthesis and characterization

Industrial research activities (R & D programs)

- Product characterization
- Thermal stability of construction materials, alloys, glass objects and packaging materials
- Metal Oxidation

Staff & Contact Information

A Scientific/Administrative Committee is responsible for the Unit. Information can be obtained by contacting Prof. T. Vaimakis:

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Representative Publications

1. G.C. Koumoulidis, C.C. Trapalis, and T.C. Vaimakis, "**Sintering of Hydroxyapatite Lath-like Powders**", *J. Thermal Anal. Calorim.*, **84**, 165 (2006).
2. T.C. Vaimakis, E.D. Economou, and C.C. Trapalis, "**Calorimetric Study of Dissolution Kinetics of Phosphorite in Diluted Acetic Acid**", *J. Thermal Anal. Calorim.*, **92**, 783 (2008).
3. N. Todorova, T. Giannakopoulou, T. Vaimakis, G. Romanos and J. Yu, C. Trapalis, "**Preparation of fluorine doped TiO2 photocatalysts with controlled crystalline structure**" *Inter. J. Photoenergy*, Art. No. 534038 2008
4. A. Giannakas, C. G. Spanos, N. Kourkoumelis, T. Vaimakis and A. Ladavos, "**Structure and Thermal Stability of Polystyrene/Layered Silicate Nanocomposites**", Composite Interfaces **16**, 237 (2009)
5. Anastasios I. Mitsionis, Tiverios C. Vaimakis, "**A Calorimetric Study of the temperature effect on Calcium Phosphate precipitation**", *J. Thermal Anal. Calorim.*, **99**, 785 (2010).
6. A.J. Mitsionis, T.C. Vaimakis, and C.C. Trapalis**, « The effect of citric acid on the sintering of the calcium phosphate bioceramics**", *Ceramic International*, 36, 623 (2010),.
7. G.I. Chilas, N. Lalioti, T. Vaimakis, M. Kubicki, and Th. Kabanos, "**Hydrothermal syntheses, crystal structures and physicochemical properties of 2-D and 3-D inorganic coordination cobalt(II)-sulfite polymers**", *Dalton Trans.*, (2010), *(accepted).*
8. K. Mahmud**,** T.C. Vaimakis, A.J. Mitsionis, and C.C. Trapalis**, "The Threonine Effect on Calcium Phosphate Preparation from a Solution Containing Ca/P=1.33 Molar Ratio**", *Ceramic International*, (2010), *(accepted).*