

**Scope of accreditation of research laboratory
No AB 120
issued by Polish Centre for Accreditation Issue
No. 17 of 12 June 2019.**

Head of the laboratory

The experts

Anna Wierzbicka, PhD, D.Sc.
a.wierzbicka@imim.pl

Tomasz Gancarz, PhD, D.Sc., Deputy Head of the Laboratory)

Tomasz Czeppe, PhD, D.Sc.
Grzegorz Garzeł, PhD, Eng.
Marek Kopyto, PhD, Eng.
Łukasz Rogal, PhD, D.Sc., Eng.
Sylwia Terlicka, PhD

Address:

**Institute of Metallurgy and Materials Science Polish
Academy of Sciences**

ul. Reymont 25, 30-059 Krakow

phone: (48) 12 295 28 98, fax: (48) 12 295 28 04

e-mail: zlb@imim.pl, website: <http://www.imim.pl>

Equipment:

1. Differential scanning calorimeter, type 910, producer: DuPont ;
2. High precision differential scanning calorimeter, type.Q1000, producer: TA Instruments;
3. Differential scanning calorimeter, type F1 404 Pegasus, producer: Netzsch Comp.;
4. Differential scanning calorimeter + thermo balance (SDT), type Q600, producer TA Instruments;
5. Thermo-mechanical Analyzer (TMA)-equipped with 1 Hz modulations, type 402 F1 Hyperion, producer Netzsch Comp.

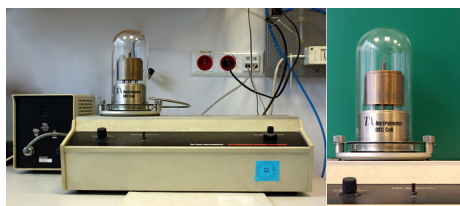
A short characteristics of the equipment:

Differential Scanning Calorimeter (DSC) is dedicated to the precise measurement of the heat consumed or released in a phase transformation versus temperature and heat capacity C_p determination outside of the temperature range of the phase transformations. All the DSC calorimeters in the Lab. are of the heat flow-type. Measurements are performed in the controlled inert gas atmosphere: helium, argon or nitrogen of high purity. The gas atmosphere may be additionally purified. The calorimeters are used both in experiments with continuous

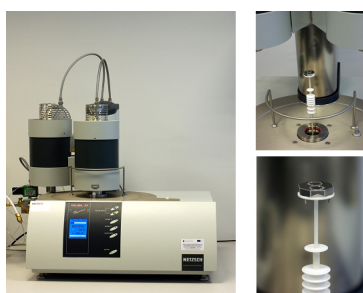
heating, isothermal and with special thermal program (e.g. wise step). Also, measurements in cooling or in thermal cycles are performed. The required precision is achieved by the proper calibration procedures, devoted to the particular type of experiment. The determination of the phase diagrams, elements of the process kinetics, purity or samples identity control are available. Also explosive materials and fuels may be characterized.



DSC Q1000 TA Instruments: a flux type calorimeter, temp. range **-170 - +400°C**, heating rates 0.5 - 50K/min, liquid nitrogen (LNCS)/compressed air cooling systems, temperature modulation technique (MDSC).



DSC DuPont 910: a flux type calorimeter of simple construction, temp. range **+25 - +700 °C**, heating rates 0.1 - 50K/min, gas nitrogen/ballistic cooling systems, controlled path for the inert gas.

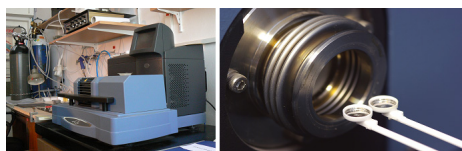


DSC 404 F1 Pegasus, NETZSCH: flux type calorimeter, temp. range **-170 - +1000 °C** (steel furnace) and **+50 - +1650 °C** (rhodium furnace), real DSC measurement. Heating rate- 0,01 - 50K/min. Different types of sample-carriers for different type of experiments. Vacuum system up to 10⁻² mbar / purge gas flux controlled. Gas type changeable during the experiment. Modulated DSC mode of operation (TM-DSC). Cp measurements in high temperature range. Reach analytical & kinetic software. Automatic system for base-line correction.

Scanning Differential Thermal and Thermo-Gravimetric Analyzer (SDT):

SDT Q 600 TA enables simultaneous precise measurement of the mass and temperature changes with a different thermal programs, that is TG +DTA+DSC methods. DSC signal is calibrated. Reactions and phase transformations including mass changes, like oxidation and decomposition may be examined in purging or reactive gas atmosphere.

Experiments both in heating and cooling modes are available. Determination of phase diagrams, T-T-T diagrams, decomposition processes, chemical reactions, oxidation and other phase transformations may be done.



SDT Q600 TA Instruments: DTA/TG applicable temp. range + 200 - +1500 °C

. Heating rates: 1 - 40K/min. Except regulated flux of purging gases application of the reactive gas is possible, gas may fulfill sample zone or may be directed on the sample. Purging / reactive gas exchange is possible during experiment.

Measurements in heating and cooling or in the thermal cycles available. Thermal program constructed with heating, cooling, isothermal, wise or other steps by operator. Kinetic software for

DSC and TG experiments including oxidation and decomposition. May be used for determination of phase diagrams, decomposition, reactions, oxidation other phase transformations.

Thermo-mechanical Analyzer (TMA) the equipment used in the thermal analysis to determine sample dimensions changes and mechanical properties, in dependence on the temperature and load. Phase transformations influencing sample dimensions are determined. Both, static and dynamic loads may be applied. Different types of loads and the possibility of modulated loads (1 Hz) enables to determine elastic constants. Measurements in the inert gas atmosphere may be performed. Typical modes of operation thermal expansion, creep, penetration, tensile and compression loads, three-point bending are available. Modulated mode with 1 Hz frequency (DMA type of measurement), sinusoidal, rectangular or other shapes of the load modulations may be applied. DTA type of additional signal is available. Analytical programs analyzing results.



TMA 402 F1 Hyperion, NETZSCH: useful sample dimensions: length 0.01 to 20 mm, diameter 1 - 10 mm. Starting length of the sample determined automatically. Load in the range $\pm 3\text{N}$, increase in 1 mN steps. Resolution of the deformation - 0.125 nm in the range of 5000 μm . Temperature range

-170 - +1000 °C

(steel furnace) and

+25 - +1550 °C

(silicon carbide furnace). Multi-point temperature calibration.

Inert gas (nitrogen, argon or helium). Vacuum up to 10⁻² mbar available, inert gas flux controlled. Operation modes: thermal expansion, creep, penetration, tensile and compression loads, three-point bending. Sample holders made with Si and Al₂O₃.

Modulated modes with 1 Hz frequency (DMA type of measurement), different shapes of the modulated loads available. DTA type of additional signal. Liquid nitrogen cooling unit. Analytical programs calculating results in reference systems: time / temperature - load, deformation - time / temperature or time / temperature-dimensions. Elastic constants, phase transformations, plastic deformation range may be determined.

Sample preparation for experiments: please contact the Laboratory.

Typical subjects analyzed in the Lab.:

- Non-diffusional phase transformations like martensitic transformation;
- Decomposition, oxidation and degradation processes;
- Diffusional phase transformations, thermal stability of materials, decomposition with gas phase participation, recrystallization;
- Experimental verification of phase diagrams;
- Glass transition, crystallization, kinetics of phase transformations, T-T-T diagrams determination;
- Heat capacity;
- Phase transformations at the grain boundaries of nanomaterials, thermal stability;
- Classes of materials: alloys, high temperature intermetallic phases, metallic glasses, polymers and plastics, ferroelectrics, soaps, emulsions.

Contact for the Client:

The Head of the Laboratory L-5:

Anna Wierzbicka-Miernik, PhD, D.Sc.

phone: **+48 12 295 28 51, 295 28 73**

e-mail: a.wierzbicka@imim.pl

PCA

Zakr

Laboratorium Kalorymetrii i Analizy Termicznej L-5 ul. Reymonta 25; 30-059 Kraków		
Przedmiot badań/wyrób	Rodzaj działalności/ badane cechy/metoda	Dokume
Elastyczny zakres akredytacji ^{1, 2, 3, 4}		
Metale, stopy metali, materiały ceramiczne i związki chemiczne i polimery ¹⁾	Temperatura, ciepło właściwe, ciepło przemian Badania kalorymetryczne różnicowe, skaningowe (DSC) ²⁾	P/19/IB-13 ⁴⁾
	Temperatura, ciepło przemian, zmiana masy, rozszerzalność cieplna, odkształcenie. Różnicowa analiza cieplna, termograwimetria wysokotemperaturowa, różnicowa kalorymetria skaningowa, analiza termo-mechaniczna ^{2) 3)}	P/19/IB-14 ⁴⁾

- 1) Dodanie przedmiotu badań w ramach grupy przedmiotów badań
- 2) Dodanie badanej cechy w ramach grupy przedmiotów badań i techniki badawczej
- 3) Zmianę zakresu pomiarowego metody badawczej
- 4) Stosowanie zaktualizowanych metod opisanych w procedurach opracowanych przez laborat

Lista działań prowadzonych w ramach elastycznego zakresu akredytacji jest udostępniana publicznie na stronie internetowej podmiot.

Laboratorium formułuje opinie i interpretacje w sprawozdaniach z badań podanych w powyższej

Laboratory of Microcalorimetry - L-5 25 Reymonta Str. 30-059 Kraków		
Investigated objects / Group of objects	Investigated features and methods of investigations/measurements	Standards and/or investigation procedures
Metals, alloys, ceramic materials, compounds, polymers	Temperature, heat capacity, transformations latent heat, Differential scanning calorimetry ¹⁾	P/19/IB-13
	Temperature, transformations latent heat, mass change, thermal expansion, deformation. Differential thermal analysis, thermogravimetry, high temperature differential scanning calorimetry, thermo-mechanical analysis ¹⁾	P/19/IB-14

¹⁾ It is allowed: implementation of new own test methods, modification of own test methods, change of measurement range of the test method, application of the updated standard methods, addition of investigated feature within the framework of object and method as well as addition of object within the framework of investigated feature and method.

The current list of tests conducted within the scope of elastic range is available on the request at an accredited entity.

The person responsible for the opinions and interpretation included in the research report, formulated on the basis of the results presented in the above table - Prof Tomasz Czeppe, Ph.D., D.Sc.

version of the page: A