DOI: 10.63527/1607-8829-2026-1-45-66

V.V. Lysko[1,2] (https://orcid.org/0000-0001-7994-6795),
O.Yu. Mykytiuk[1,3] (https://orcid.org/0000-0001-9365-4836)

[1]Institute of Thermoelectricity of the NAS and MES of Ukraine,
1 Nauky str., Chernivtsi, 58029, Ukraine;
[2]Yuriy Fedkovych Chernivtsi National University,
2 Kotsiubynsky str., Chernivtsi, 58012, Ukraine;
[3]Bukovinian State Medical University,
2 Theater Square, Chernivtsi, 58002, Ukraine

Corresponding author: O.Yu. Mykytiuk, e-mail: *orusia2@gmail.com*

Application of Microcalorimetry in Materials Science

This review article discusses the practical use of calorimetric methods in materials science, in particular for studying phase transitions, determining heat capacity and thermal properties, investigating defects and structural changes, studying polymers, biomaterials, surface phenomena, etc. The application of microcalorimetry for monitoring explosives and pyrotechnics is highlighted.

Keywords: microcalorimetry, materials science, phase transitions, polymers, biomaterials, explosives.

Introduction

Microcalorimetry is an extremely powerful tool for studying the properties of materials, which allows researchers to quantitatively assess the energy changes that accompany physical, chemical and structural processes in various materials and provides unique information about the structure, stability and kinetics of transformations. Due to its high sensitivity, it allows studying subtle thermal phenomena that are difficult or impossible to investigate by other methods.

As a result of physicochemical processes in the sample under study, which is located in the reaction chamber, heat flows arise. The operation of existing microcalorimetric measuring sensors, which convert the temperature of the reaction chamber or the heat flow arising in it into an electrical signal, is based on physical phenomena of different nature. Thermoelectric phenomena also belong here [1].

Citation: V.V. Lysko, O.Yu. Mykytiuk (2026). Application of Microcalorimetry in Materials Science. *Journal of Thermoelectricity*, (1), 45–66. https://doi.org/10.63527/1607-8829-2026-1-45-66

Received: 02.02.2026; Revised: 27.02.2026; Published: 31.03.2026

In many microcalorimetric measurement methods, active thermal effects on the reaction chamber and other structural elements are used, which are controlled by the introduction or withdrawal of a certain amount of heat for thermostating or compensation of thermal effects of the object under study. For these purposes, such thermoelectric phenomena as the longitudinal Seebeck effect, longitudinal and transverse Peltier effects are used. Vortex thermoelements, which are based on the effect of eddy thermoelectric currents, are also used as calorimetric sensors [2, 3].

The use of thermoelectric sensors or thermopiles allows the measurement of very small heat flows (on the order of microwatts and less), which is critical for studies of small samples. In differential microcalorimeters (e.g., Calvet or DSC types), thermoelectric sensors are used to measure the difference in heat flows between two identical chambers: one containing the sample and the other containing the reference. This allows compensation for background noise and temperature fluctuations, significantly increasing the accuracy of the measurements [4, 5].

Calorimetric methods are used to study temperature, heat and kinetics of phase transitions, to determine heat capacity, thermal diffusivity and thermal conductivity, which is important for simulation of heat transfer. The study of chemical reactions in materials by the calorimetric method allows us to determine kinetic parameters, study the stability of materials and surface phenomena. By measuring heat flows, microcalorimetry makes it possible to study defects and structural changes in various materials, to study polymers, biomaterials and soft materials, and pharmaceutical products [6,7,8].

Microcalorimetry is widely used to control explosives and pyrotechnics, in particular to assess their thermal stability and durability, evaluate the compatibility of components, control the quality of the substance and the resulting product, study the kinetics of the combustion process and the initial stage of the explosion, and develop new explosives.

In this paper, we will examine how microcalorimetry helps understand the behavior of materials by measuring thermal effects (heat release or absorption), which directly result from physical and chemical processes occurring at the molecular and atomic levels. The resulting experimental data reveals the bond energy between atoms and molecules, a key aspect of microstructure and opens up new possibilities for creating materials with improved properties.

I. The main types of calorimeters that are used in materials science

In modern materials science, various types of calorimeters are used. The range of temperatures studied by modern calorimeters is 0.1÷4000 K, the values of the measured amount of heat are in the range from 10^{-5} to several thousand J, the accuracy of the results is estimated as 10^{-2} %. The duration of the studied processes is in the range from fractions of a second to tens of days.

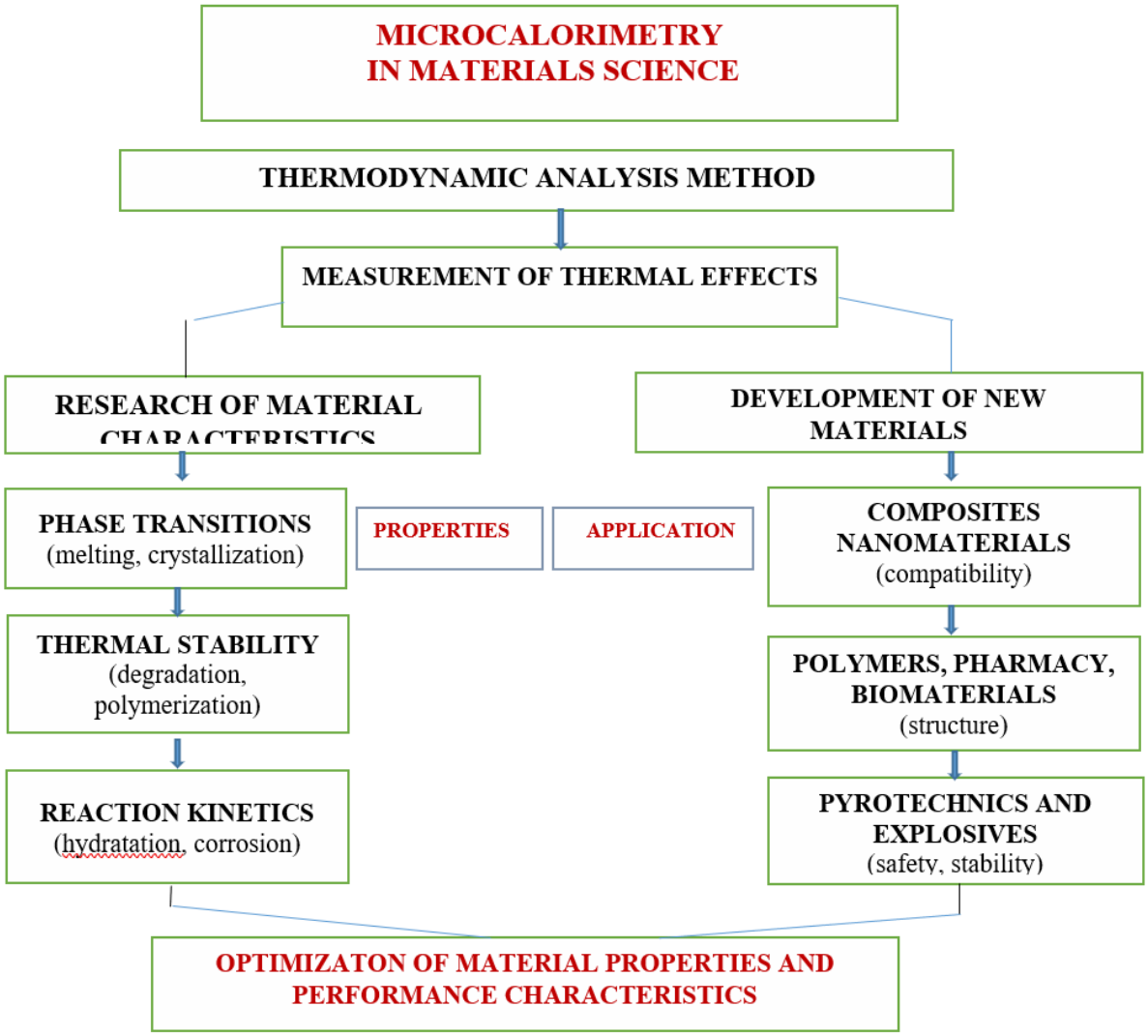
The main types of calorimeters that are widely used in materials science are presented in the following table.

Type of calorimetry	**Abbreviated name**	**Basic measurement mode**	**What is measured (Key parameters)**	**Main areas of application in materials science**
Differential scanning calorimetry	DSC	Dynamic (temperature change over time)	Temperature and enthalpy (ΔH) of phase transitions (glass transition, melting, crystallization) and specific heat (C_p)	*Polymers:* T_g (glass transition temperature, T_m (melting temperature), degree of crystallinity. *Металл***:** Phase diagrams, thermal stability of alloys. .
Isothermal microcalorimetry	ITC/TAM (Isothermal Titration/ Thermal Activity Monitor)	Isothermal (constant temperature)	Thermal flow (Q) as a function of time. Determination of kinetics and thermal effect (Q).	*Reaction kinetics*: Polymerization, curing, cement hydration, corrosion. Ageing/stability: Life prediction (e.g., pharmaceuticals, explosives, biomaterials).
Heat flow calorimetry	HFC	Isothermal or dynamic	The amount of heat released or absorbed during a reaction.	*Chemical safety*: Study of exothermic reactions (thermal acceleration). Chemical engineering: Optimization of industrial processes.
Bomb calorimeter	BC	Adiabatic/ Isoperibolic	Heat of combustion (energy value) (ΔU, ΔH).	*Fuel and energy*: Determination of the calorific value of coal, petroleum products, biofuels. Explosives: Estimation of energy content

DSC is an indispensable tool for rapid qualitative and quantitative analysis of structural changes in a material upon heating or cooling. ITC/TAM microcalorimetry is a method extremely important for kinetic analysis, measuring very small thermal effects that occur slowly at a constant temperature and providing information critical for predicting the service life of a material. Heat flow calorimetry is a set of methods for measuring the amount of heat released or absorbed in a process (physical, chemical or biological), as well as measuring the heat flow

itself. These calorimeters are equipped with heat flow sensors (e.g. thermoelectric modules) that measure heat output. The bomb calorimeter is indispensable for analyzing the energy value of fuels, food products, pyrotechnics and explosives.

II. Schematic analysis of the main areas of application of microcalirimetery in materials science:



III. Examples of practical applications of microcalorimetry in materials science

1. Study of phase transitions

Phase transition temperatures: precise determination of melting, crystallization, glass transition, polymorphic transformation temperatures, the Curie effect, etc. This is important for metals, alloys, polymers, ceramics, and composites. For example, the Curie temperature and phase transition heat were studied in $Ba_xSr_{1-x}TiO_3$materials [9]. Certain parameters were verified either by the temperature dependence of the permittivity or by the thermodynamic method. Microcalorimetry was found to be a useful tool for studying phase transition

phenomena in ferroelectric perovskites. In [10], granular microcapsules with BASF Micronal DS5038 X phase change material (PCM) were studied using thermal analysis: thermogravimetry (TG) and DSC in the temperature range from –20 °C to 55 °C or 80 °C, which corresponds to two separate phases of the filler phase transition. Heating/cooling rates from 1 to 10 K min^{-1} were used. The enthalpy, the onset/end temperatures of the phase transitions and the temperature dependences of the specific heat capacity were determined.

Phase transition heats: Quantifying the enthalpy of phase transitions provides insight into the energetics of the process, which is important for thermodynamic modeling and predicting the behavior of materials in different situations. Nanoscale methods such as small-angle neutron scattering, supplemented by microcalorimetry to determine the phase composition and domain structure, are used to detect phase separation in membranes. The coexistence of solidus-liquidus phases in a mixture of saturated lipids was investigated in [11], which showed an overlap of the boundary traces for multilamellar and unilamellar 50 nm vesicles.

Phase transition kinetics: Studying the rates and mechanisms of phase transformations (such as polymer crystallization or quenching/tempering of metals) enables optimization of materials processing. Phase transitions are a key factor in changing the mechanical or electronic properties of materials. Understanding the thermodynamics and kinetics of these transitions forms the basis of modern materials science. Phase transition kinetics are studied by measuring heat flow, and microcalorimetry allows for real-time monitoring, providing insight into the energetics of the process.

The kinetics of the α⇔β phase transformations in the Ti–4.4 wt.% Ta–1.9 wt.% Nb alloy was studied in [12] using isochronous DSC in the range of heating/cooling rates 3–99 °C·min^{-1}. It was found that the α→β transformation is diffusion-controlled, and the reverse is controlled at the interface. The determined thermokinetic parameters allowed us to construct continuous heating/cooling diagrams, which is useful for selecting the parameters of production and heat treatment of the alloy. In [13], the ferroelectric phase transition in barium titanate under pressure was investigated using DSC and Landau-Ginsburg theory, demonstrating an innovative method of thermal measurements. A new tricritical point characterizing the change in the phase transition was experimentally determined and theoretically confirmed. Increasing the depth of quenching expands the pressure range for the multidomain structure and narrows it (up to disappearance at p = 145 MPa, T = 395 K) for the single-domain one. That is, the pressure during quenching can be used to control the physical properties of the perovskite material. In [14], the behavior and kinetics of the phase transition of zirconium alloys under non-isothermal conditions were analyzed using DSC. The phase transition temperature was studied on DSC curves. Its dependence on the heating rate was revealed. The change in enthalpy of 1035 steel during phase transformation at different cooling rates was also measured using DSC [15]. The results showed that the activation energy of the phase transformation process varied depending on the transformation fraction, and the functions of the transformation process mechanism differ in different temperature ranges.

Simulation using the JMAK equation [16] showed a good agreement with the DSC data of the kinetics of martensite-austenite transformation in high-temperature Ni-Ti-Hf shape

memory alloys obtained at four heating rates. It was found that the heating rate regulates the transformation enthalpies, allowing control of the phase transition parameters. In the study [17], cylindrical rods made of martensitic-aging Fe-Cr-Ni-Al steel, manufactured by laser powder deposition, were analyzed by DSC. During continuous heating at different rates, heat flux peaks were identified that corresponded to the phase transformations of precipitation and austenite reversion. The temperatures of the beginning and end of these transformations were determined.

2. Determination of heat capacity and thermal properties

Specific heat capacity (C_p). Accurate measurement of the specific heat capacity of materials over a wide temperature range is important for heat transfer calculations, thermal system design, and simulation of material behavior.

*Thermal diffusivity and thermal conductivit*y. Direct measurement of heat capacity and heat flows can be used in combination with other methods for determining thermal conductivity.

In [18], using an improved RD496-III microcalorimeter, a formula was developed to determine the specific heat capacity of thirteen solid compounds with a total deviation within 1.0 %. A special calorimetric device was developed to measure the specific heat capacity of asphalt concrete [19].

In [20], a method of continuous thermal pulse measurement for the simultaneous analysis of heat consumption Q and heat capacity C_p in liquids is presented, which allows overcoming the difficulties associated with fluctuations in *Cp* during water evaporation. The method consists in directly applying a 400 nl drop of sample to a microcalorimeter, which is repeatedly heated by light-emitting diode (LED) radiation (100 ms pulses, 10 s repetition), with simultaneous measurement of the temperature response. In [21], a new approach is presented for more accurate (±1−2%) and reproducible measurement of the specific heat capacity C_p (J K^{-1} g^{-1}) of materials using DSC.

3. Study of chemical reactions in materials

Calorimetry is also used to measure enthalpy changes during chemical processes. The calorimetric method is used to study: 1) reaction kinetics - measurement of thermal effects accompanying chemical reactions (polymerization, decomposition, oxidation, corrosion); 2) stability of materials – studying the thermal stability of materials can reveal the onset of degradation, which is accompanied by the release or absorption of heat; 3) surface reactions - studying the adsorption/desorption of gases or liquids on the surfaces of materials (for example, in catalysts, sorbents) to obtain information about the binding energy and interaction mechanisms.

A new setup for heat analysis of continuous single- and multiphase reactions is presented in [22]. The principle is based on the measurement of real heat flow with a resolution of 10 mW. In addition to mixing ethylene glycol with water, industrially significant exothermic processes were investigated: phenol nitration, nitrobenzene reduction, and other redox reactions. The commercial ChemiSens CPA202 calorimeter was modified with a glass static mixer and a

microreactor, allowing volumes up to 5.5 ml. The experiments were carried out by feeding separate streams of liquid or gas using pumps, mixing them in a thermostated zone of the calorimeter.

The method of calorimetric measurements of temperature changes caused by heat exchange between objects or substances during a chemical reaction is described in the book [23]. Methods for measuring enthalpy changes ΔH of chemical processes are presented. Heat flow measurements can be made using a constant pressure calorimeter (directly obtaining ΔH) or a bomb calorimeter (for combustion enthalpies at constant volume).

Paper [24] reports the development of calorimetric sensor arrays capable of measuring nanometer-thick samples at rates up to 105 K/s, suitable for the study of complex materials using a combinatorial approach based on sputtered thin films and computational modeling. Nanocalorimetry is ideal for assessing the kinetics of solid-state and gaseous reactions, and its use in multilayer Zr/B and Zr/B_4C materials is demonstrated. The capabilities of a new isothermal calorimetric sensor are demonstrated for use in biophysics in studying the energetics of an active microtubule gel driven by molecular motors.

The simultaneous determination of the enthalpy of mixing and reaction in a milliscale continuous flow calorimeter was investigated in [25], which is important for assessing the safety of chemical processes. Due to the increasing demand for lithium-ion batteries and the risk of their thermal explosion, the decomposition kinetics are studied by the calorimetric method [26]. Experimental data improve the approximation models of the process.

4. Study of defects and structural changes

Crystal lattice defects are determined by measuring the energy associated with the formation, migration, or annihilation of point defects (vacancies, interstitials) and dislocations in crystalline materials. *Aging processes* are studied by studying the subtle thermal effects associated with the aging of materials (e.g., polymers, alloys), which can indicate changes in their structure and properties [27]; *Radiation defects* are detected by studying the energy released or absorbed during the formation and annealing of radiation defects in irradiated materials, which is critically important for nuclear power.

The availability of high-temperature Calvet calorimeters, improvements in thermal analysis equipment, and the possibility of accurate cryogenic measurements of the heat capacity of milligram samples were noted in [28]. The study of refractory ceramic materials is complicated by their non-reactivity, complex structures, multicomponent composition, non-stoichiometry, and order-disorder phenomena.

For refractory ceramics and minerals, the use of molten oxide solvents at 700–800°C is effective. In [29], the Wadsley-Roth phase shift compound $(W_{0.2}V_{0.8})_3O_7$, a promising material for fast-charging electrodes, was studied by operando calorimetry. Calorimetric analysis showed that the synthesized nanoparticles have lower ionic resistance, are able to accommodate more lithium, and lithium intercalation in them is kinetically more favorable.

The kinetics of amorphous defect phases localized at grain boundaries using ultrafast DSC at high temperatures and scan rates was investigated in [30]. The materials were

purposefully treated by annealing/quenching. The understanding of grain boundary processes was deepened, which has potential significance for the design and optimization of advanced materials. In [31], for highly accurate and efficient characterization of thermophysical parameters associated with martensitic transformation in shape memory alloys, it was proposed to use modulated DSC, which allows detecting the reversible and irreversible parts of thermal events during MP and obtaining useful thermal parameters.

5. Research on polymers

Microcalorimetry is a valuable method for studying polymers, providing insight into their energetics and interactions. It allows us to study phenomena such as polymer adsorption, phase transitions, and the influence of additives, offering a detailed understanding of the behavior of polymers. In particular, determining the glass transition temperature, which is an important characteristic of amorphous polymers; studying the processes of crystallization, melting, polymorphic transitions and their kinetics in crystalline and semi-crystalline polymers; monitoring the thermal effect of polymerization, which allows us to control the process and optimize synthesis conditions; detecting thermal effects of mixing or phase separation in polymer mixtures.

In [32], an integrated microfluidic thermal sensor for the characterization of the thermal properties of nanoliter liquids and thin polymer films is presented. The device, consisting of a polysilicon heater and microthermopiles on a thermally insulated membrane, allows for alternating current calorimetric measurements to determine the thermal conductivity of liquids and five typical polymers. In [33], ITS experiments are presented for measuring the heat of adsorption of polyethylene glycol on nonporous silica nanoparticles, which allowed determining the adsorption isotherm and revealing the influence of temperature and molecular weight on the interaction parameters. In [34], the latest developments in the application of DSC for the physicochemical analysis of polymeric materials are highlighted.

The problem of slow degradation of plastic and non-biodegradability of organic compounds is a significant environmental risk. In [35], microcalorimetric analysis showed that 1 % rosemary extract increases the biodegradability of polyethylene films, reducing the degree of their crystallinity. With the development of additive manufacturing, it is relevant to expand the library of materials. In laser powder deposition, where thermoplastics are used, powdered thermosetting polymers can also be used. The problem of slow plastic degradation and the non-biodegradability of organic compounds is a significant environmental risk. In [35], microcalorimetric analysis showed that 1% rosemary extract increased the biodegradability of polyethylene films by reducing their crystallinity. With the development of additive manufacturing, expanding the material library is essential. Laser powder cladding, which uses thermoplastics, can also utilize powdered thermosetting polymers.

To assess their compatibility, [36] compared the curing behavior of a commercial polyester powder coating as a model material using DSC and fast scanning calorimetry (FSC) at heating rates from 5 to 7500 °C/min. Both methods revealed curing exotherms. [37] studied the thermal properties and pyrolysis decomposition of cotton fibers modified by silanization

and sulfation-phosphorylation using various methods, including DSC. The results are important for the application of modified cotton fibers in fire-resistant materials.

Microcalorimetry and thermal analysis were used to study BND 70/100 bitumen and polymer-modified bitumens based on it. The influence of individual polymers on the structural organization of bitumen and its resistance to thermal degradation was revealed, which has a significant impact on operational characteristics [38].

6. Study of adsorption and surface phenomena

Adsorption microcalorimetry - measurement of the heat of adsorption of gases or liquids on the surfaces of materials, which provides information about the nature and strength of the bond between the adsorbate and the surface, as well as the number of active centers. The study of the thermal effects of surface wetting by liquids is important for understanding adhesion, material compatibility, and coating formation processes.

Review [39] highlights the key role of microcalorimetry in the study of solid-liquid interactions. Microcalorimetry provides important thermodynamic information that helps to understand the direction and limitations of interactions. Common methods such as DSC, ITC and immersion microcalorimetry are reviewed. The factors affecting the enthalpy change and the specific applications of microcalorimetry in the study of various solid-liquid binding processes are discussed and it is noted that a significant amount of information on solid-liquid interactions still awaits investigation using calorimetry.

In [40], the textural parameters of graphene oxide (GO) and graphite (Gr) samples were determined. The pore size distribution was estimated using mathematical modeling. The results were compared with the immersion enthalpies obtained using molecules of different kinetic diameters (0.272–1.50 nm). It was shown that the calorimetric method provides results comparable to the data of N_2 adsorption isotherms at 77 K.

Due to the need to reduce CO_2 emissions, layered double hydroxides and their derivatives are being investigated as promising CO_2 adsorbents due to their low cost, easy synthesis, high sorption capacity and surface basicity. Adsorption calorimetry [41] allowed us to establish a linear correlation between the surface basicity of the obtained mixed oxides and their sorption capacity for CO_2.

The curing of cyanoacrylate adhesive was investigated using a microcalorimeter. The effects of gap formation and adsorption of an acid stabilizer of the adhesive by various metal and glass substrates were studied [42].

7. Research into biomaterials and soft materials

Microcalorimetry is a powerful method for studying both biomaterials and soft materials due to its ability to measure the heat flow associated with various processes, which provides insight into materials properties, molecular interactions, and stability over time.

Interaction of biomolecules with materials. Studying the thermal effects of interactions of proteins, nucleic acids, or lipids with biomaterial surfaces is crucial for the development of

implants, drug delivery systems, and biosensors. ITS is a key method for understanding the interactions of biomolecules and nanoparticles, which is fundamental for biomedical, environmental, and toxicological applications [43]. ITS quantitatively determines the thermodynamic parameters of intermolecular interactions in situ, providing information on the binding affinity, interaction mechanism (ΔH, ΔS, ΔG), and binding stoichiometry. This improves the mechanistic understanding of the protein corona around nanomaterials in biological environment. A mini-review [44] shows that nanomaterials with a hydrophilic surface, without a strong charge and with steric stabilization, exhibit the weakest and least nonspecific interactions with proteins, which makes them the most promising for the formation of a controlled protein corona. The potential application of ITS and flow calorimetry to study specific problems and relationships of the adsorption behavior of proteins and various factors affecting it is studied in [45, 46]. Review [47] aims to deepen the understanding of biomolecular interactions by using the ITC method for the thermodynamic characterization of two important biomaterial systems: self-assembling peptides and non-fouling polymer-modified surfaces. Understanding the molecular basis of specificity and recognition between proteins and ligands is a key goal of modern molecular biology. The most accessible thermodynamic quantity for a protein entering a bound state is the enthalpy [48].

Stability of biomaterials. Provides for monitoring the thermal stability of biomaterials, hydrogels, liposomes. Report [49] discusses the application of quasi-isothermal modulated differential scanning calorimetry (QiMDSC) for the analysis of soft tissue biomaterials and their crosslinking mechanisms, in particular using glutaraldehyde (Glut). QiMDSC, a variation of traditional DSC, is already used to analyze polymorphic transformations in the pharmaceutical and food industries.

The book “Biomaterials: From Carousel Material Design to Implant Optimization” presents the use of DSC in research related to biomaterials and their modifications [50]. In particular, the research concerns: 1) chemically induced crosslinking of peptide fibrils to create scaffolds of polymer particles and macrophages; 2) studying a coating consisting of a thiol-terminated self-assembled monolayer and immobilized low molecular weight heparin (LMWH) to locally prevent blood clot formation on the surface of titanium dioxide (TiO_2) in the motor of a left ventricular assist device; 3) optimizing a material for intraocular lenses with a high refractive index and hydrophobicity.

8. Control of pyrotechnic and explosive substances by microcalorimetry

Microcalorimetry is an important tool for quality control, safety and stability of pyrotechnic and explosive substances. It allows you to study the thermal changes that occur in these substances during chemical reactions and phase transformations and obtain critically important information. Advantages of microcalorimetry for explosives control: 1) high sensitivity, i.e. the ability to detect even minor thermal changes, which is especially important for monitoring slow decomposition or interaction processes; 2) small sample sizes – very small amounts of substance (milligrams) are analyzed, which increases the safety of experiments; 3) controlled conditions – the ability to conduct measurements under conditions of precisely

controlled temperature, pressure, gas environment; 4) versatility – the use of different measurement modes (isothermal, dynamic).

Let us consider the main applications of microcalorimetry in the control of pyrotechnic and explosive devices.

1. *Thermal stability and durability assessment* consists in determining the exothermic decomposition temperature, which is a key indicator for assessing the stability and safe storage of explosives. Kinetic parameters (activation energy, pre-exponential factor) of decomposition reactions are obtained by isothermal or dynamic measurements, which allows predicting the service life and conditions of safe storage of materials. Studies of explosive samples at elevated but safe temperatures make it possible to simulate aging processes and assess their impact on stability and properties. The presence of small amounts of unstable impurities can significantly affect the thermal stability of explosives. Microcalorimetry can detect these anomalies in the thermal profile.

2. *Assessing component compatibility:* Pyrotechnic and explosive mixtures often consist of several components. Microcalorimetry allows for the detection of undesirable chemical reactions or physical interactions between them. Assessing storage safety involves identifying exothermic reactions between components long before dangerous temperatures occur, allowing for risk assessment and safer storage of explosives.

3. Quality control of raw materials and finished products: each explosive material or pyrotechnic component has a unique thermal fingerprint (melting point, decomposition temperature, phase transition temperature). This allows the use of microcalorimetry for rapid identification and verification of raw material purity; comparison of the thermograms of the samples being tested with reference data allows for verification of product compliance with established quality and safety standards.

4. *Studying combustion and explosion kinetics (early stages):* Although microcalorimetry does not measure the detonation rate itself, it can provide data on the initial, slower reactions that precede combustion or explosion.

5. Development of new explosive formulations. Thermal effects studies allow us to evaluate the influence of various additives, binders, or modifiers on the stability and thermal behavior of new explosive formulations and assist in studying the energetic aspects of chemical transformations in explosive formulations.

Paper [51] is an introduction to thermal analysis (including DSC) of explosives and powders, covering parameters such as heat capacity, mass loss, reaction onset temperatures and enthalpies. Within the framework of the A95KL486 project (at the request of DMKL), a database of explosive and propellant characteristics was created. For this purpose, 16 explosives, 5 propellants and 4 polymers were investigated using DSC and DTA/TG. Key parameters (heat capacity, mass loss, onset temperature, enthalpies) were determined, which are a guideline for quality control of new samples and can be used as input data for modeling other experiments. DSC and TG can also be used to determine the compatibility of explosives with various polymers in accordance with STANAG 4147, one of the military standards of NATO member countries, which is limited to studying the chemical compatibility of

ammunition components with explosives and does not cover procedures and requirements for preventing non-compliance for other reasons [52].

The condition of the powder is monitored by studying its thermal behavior or determining the amount of stabilizer remaining. This is especially important for nitrocellulose-based powders, which are unstable. The causes of accidents are often old material, extreme storage conditions, or a combination of these factors. Modern test methods strive to reproduce real storage conditions as accurately as possible. For such studies, the optimal method is heat flow calorimetry [53]. Paper [54] is devoted to the study of the thermal decomposition of black powder using various thermal methods, including DSC. Exothermic reactions were observed at temperatures up to 230 and 140°C in inert and oxidizing media, respectively. In [55], a Calvet microcalorimeter was used to study the thermal behavior of a composite modified dibasic powder with RDX. The use of microcalorimetry for testing the compatibility of explosives and other materials, as well as for testing the stability of pyrotechnic materials under various conditions, is discussed in detail in [56, 57, 58]. Microcalorimetry has reportedly been used in Bofors explosives laboratories for over 10 years, mainly for testing the compatibility of explosives and other materials, as well as for testing the storage stability of pyrotechnic articles under various environmental conditions.

In [59], the chemical compatibility of two types of fuel with two types of polymer materials was investigated using heat flow calorimetry, DSC and other methods (according to STANAG 4147). Heat flow curves for fuels, polymers and their mixtures were determined and compared, the energy produced was calculated and absolute and relative compatibility indices were established. The degree of chemical instability strongly depends on the chemical structure of the explosives - aromatic and aliphatic nitro compounds, secondary nitramines and organic azides are relatively stable, while aliphatic nitrate esters have a much lower stability. The aging rate of explosives can be significantly accelerated by incompatibility reactions between explosives and contact materials [60].

Compatibility is critical for energetic materials and their additives (casings, binders), as incompatibility creates additional risks during ammunition handling and storage. Several compatibility tests were compared in accordance with NATO standards. A wide range of energetic materials (single- and dual-base propellants, explosives: RDX, PETN, HMX, TNT) and additives (Teflon, polypropylene, self-igniting casing, inhibitors) were examined using various methods, including DSC. These studies are a key to safety and service life [61].

The use of DSC to measure the thermal decomposition characteristics of high-energy materials and obtain kinetic parameters is shown in [62]. The microcalorimetric method for testing the compatibility of components of combustible materials and cast composite explosives is based on the change in power when measuring separately different contacting materials and their mixtures during isothermal treatment [63]. The analysis of military explosives in terms of thermal stability depending on their composition is considered in [64]. In [65], the use of DSC to study the compatibility of individual explosives with different polymeric materials is shown, being an important aspect for safe storage and use, and the STANAG4147 standard was used as a criterion to assess the compatibility between the observed materials.

Paper [66] presents the use of DSC to assess the thermal hazards of nitroalkanes, a class of compounds related to explosives. Correlations of DSC data with the potential for explosion propagation are also discussed. The use of DSC to study the shelf life of energetic materials using dynamic DSC thermograms, which is a faster and more economical alternative to standard aging methods, is reported in [67]. Review [68] discusses the use of DSC to study the thermal properties of energetic materials, including decomposition and kinetics, and the problems that can arise in interpreting the data. Paper [69] using DSC, fills a gap in the knowledge of the aging effects of explosives due to low doses of radiation and is critical to establishing the safety of their handling after exposure to ionizing radiation. DSC for all samples showed no significant changes after irradiation.

Paper [70] is an example of the study of new nanoenergetic composites, where various methods, including DSC, are used to study thermal decomposition and kinetics. It is shown that the developed nanoenergetic composite based on the nitrated cellulose nanostructure can serve as a promising candidate for practical application in solid rocket fuels and composite explosives. Paper [71] discusses the standardization of thermal decomposition temperatures using DSC at different heating rates of the energetic material. The DSC methodology is central to the study, which makes it relevant for understanding modern approaches.

Pyrotechnic compositions are energetic materials made of a reactive combination of granular reducing agents with granular oxidizing agents. Due to new international standards such as REACH, many components of pyrotechnic compositions need to be replaced, so it is necessary to investigate the interaction between the components. Papers [72, 73] concern the study of important parameters of the characteristics of a given pyrotechnic composition by the DSC method. The thermal and shock sensitivity of pyrotechnic compositions containing potassium perchlorate, aluminum and graphite was studied by a group of researchers [74]. DSC and TGA were used simultaneously to study thermal sensitivity. The chemical reactions of pyrotechnics generate a large amount of heat in a closed system and lead to a thermal explosion. Although there are many thermal measurement methods to characterize the hazardous nature of pyrotechnic mixtures, accelerating rate calorimetry (ARC) is the only adiabatic and universal calorimetry that provides reliable data. ARC data can be used to establish temperature and pressure limits for the safe operation, storage, and transportation of pyrotechnics [75, 76, 77, 78].

The study of ionic liquids using thermal analysis tools, namely thermogravimetric analysis and DSC, is reported in [79]. Their thermal stability is an important factor in many potential applications, such as heat transfer fluids, battery electrolytes, and high-temperature lubricants.

In [80], the construction of a microcalorimeter of the RD496-II type for measuring the thermal conductivity of fuel pellets and explosives is reported. Two constants of the device and the thermal conductivity of seven materials were determined. Isothermal microcalorimetry was used to determine the chemical compatibility of ammunition components with explosives and rocket fuel, the fuel was studied for 168 hours at a temperature of 85 °C [81]. Paper [82] demonstrates the direct application of isothermal microcalorimetry to study the effect of temperature and stabilizers on heat generation in powders, which is critical for assessing their

durability. The two-component pellet fuel K 6210 was studied using the isothermal microcalorimetry method depending on different methods of sample preparation and the atmosphere above the fuel [83].

The aging behavior of a pyrotechnic mixture of magnesium and potassium nitrate was investigated at 50 °C and 65 % relative humidity using isothermal heat flow calorimetry [84]. Measurements were performed in air and in an inert environment. The effect of the aging process on the pyrotechnic response was also investigated using high-temperature DSC under ignition conditions and modulated temperature DSC.

Calorimetry is also used as a non-destructive analysis method to determine the output power of heat-generating nuclear materials, etc. [85], to measure the rate of nuclear heating inside a reactor [86, 87].

It should be noted that microcalorimetry, although powerful, is not the only method for controlling explosives and pyrotechnics. It provides information on the thermodynamic and kinetic aspects of their behavior, but does not replace tests for sensitivity to impact, friction, spark, as well as direct tests for detonation characteristics. Microcalorimetry is part of a comprehensive approach to assessing the safety and properties of pyrotechnic and explosive materials.

Conclusion

Microcalorimetry is a highly sensitive analytical technique that measures very small heat changes that accompany chemical and physical processes in real time and is an indispensable tool for fundamental and applied research in materials science, providing unique thermodynamic and kinetic information about the behavior of materials. This information allows researchers to gain deep knowledge about the properties of materials, their stability, interactions and reactivity. Microcalorimetry allows us to understand the energetic nature of processes in materials, control their properties and develop new compounds, alloys, polymers and nanostructures with predictable characteristics. As the capabilities of microcalorimeters improve, this field will expand, flourish and continue to prove its usefulness.

Authors' information

V.V. Lysko – Candidate of Physical and Mathematical Sciences, Acting Director of the Institute of Thermoelectricity.
O.Yu. Mykytiuk – Candidate of Physical and Mathematical Sciences, Associate Professor at the Department of Medical and Biological Physics and Medical Informatics.

References

1. Anatychuk, L.I. (1998). Thermoelectricity. Vol.1. Physics of Thermoelectricity. Kyiv, Chernivtsi: Institute of Thermoelectricity. 376 P
2. Anatychuk, L.I. (2003). Thermoelectricity. Vol. 2. Thermoelectric power converters. Kyiv, Chernivtsi: Institute of Thermoelectricity. 348 P.

3. Anatychuk, L.I. (2007). Current status and some prospects of Thermoelectricity. J. Thermoelectricity, 2, 7 – 20.
4. Kobylianskyi, R., Lysko, V., & Boychuk, V. (2024). Computer-aided design of thermoelectric microcalorimetric sensors. *Journal of Thermoelectricity*, (1-2), 97–112. https://doi.org/10.63527/1607-8829-2024-1-2-97-112.
5. Schubert, F., Gollner, M., Kita, J., Linseis, F., & Moos, R. (2016). Optimization of a sensor for a Tian–Calvet calorimeter with LTCC-based sensor discs. *J. Sens. Sens. Syst.*, *5*(2), 381–388. https://doi.org/10.5194/jsss-5-381-2016.
6. Kobylianskyi, R., Prybyla, A., Konstantynovych, I., & Boychuk, V. (2022). Results of experimental research on thermoelectric medical heat flow sensors. *Journal of Thermoelectricity*, (3-4), 68–81. https://doi.org/10.63527/1607-8829-2022-3-4-68-81
7. Anatychuk L., Kobylianskyi, R., Lysko, V., Prybyla, A., Konstantinovych, I., Kobylyanska, A., Boychuk, V. (2023). Method of calibration of thermoelectric sensors for medical purposes. *Journal of Thermoelectricity*, (3), 37–49. https://doi.org/10.63527/1607-8829-2023-3-37-49.
8. Kobylianskyi , R., Lysko, V., Prybyla, A., Konstantynovych, I., Kobylianska, A., Bukharaeva, N., & Boychuk, V. (2023). Technological modes of manufacturing medical purpose thermoelectric sensors. *Journal of Thermoelectricity*, (4), 49–63. https://doi.org/10.63527/1607-8829-2023-4-49-63
9. Radecka, M., Rekas, M. (2007). Microcalorimetry studies of phase transitions in $(Ba_xSr_{1-x})TiO_3$. Journal of Thermal Analysis and Calorimetry. 88. 10.1007/s10973-006-8131-z.
10. Omen, Ł, Panas, A J., Szczepaniak, R., Dudziński, A. Microcalorimetric analysis of granular microencapsulated phase change material including the effects of thermal cycling. Thermochimica Acta, Volume 748, June 2025, 180001. https://doi.org/10.1016/j.tca.2025.180001.
11. Krzyzanowski, N., Porcar, L, Perez-Salas, U. A Small-Angle Neutron Scattering, Calorimetry and Densitometry Study to Detect Phase Boundaries and Nanoscale Domain Structure in a Binary Lipid Mixture. *Membranes* 2023, *13*(3), 323; https://doi.org/10.3390/membranes13030323.
12. Behera, M., Raju, S., Mythili, R. *et al.* Study of kinetics of α⇔β phase transformation in Ti–4.4 mass% Ta–1.9 mass% Nb alloy using differential scanning calorimetry. *J Therm Anal Calorim* 124, 1217–1228 (2016). https://doi.org/10.1007/s10973-016-5258-4
13. Mazur, O., Tozaki, K., Yoshimura, Y., & Stefanovich, L. (2022). Influence of pressure on the kinetics of ferroelectric phase transition in $BaTiO_3$. *Physica A: Statistical Mechanics and its Applications*, *599*(C), 127436. https://doi.org/10.1016/j.physa.2022.127436
14. Liu, Jin; Liu, Yan; Deng, Ai-Lin; Chen, Hui; Cheng, Zhu-Qing;Zhang, Li; Phase transition temperature and non-isothermal kinetics in zirconium alloys by differential scanning calorimetry. Materials Express, Volume 13, Number 2, February 2023, pp. 387-394(8). https://doi.org/10.1166/mex.2023.2332

15. Tang, D., Zhang, Q. Q., Wang, F. D., Tan, G. L., & Xu, C. J. Analysis of Transformation Kinetics of 1035 Steel at Different Cooling Rates. METALURGIJA, 62 (2023), 2, 204-206. https://hrcak.srce.hr/file/419876
16. Brito, G. R. F., Castro, W. B., & Soares, R. L. (2024). Effect of Heating Rate on the Kinetics of Martensitic Transformation of Ni-Ti-Hf alloys using Differential Scanning Calorimetry (DSC). *Mat. Res. 27*. https://doi.org/10.1590/1980-5373-MR-2023-0426
17. Fabian, R., & Hadadzadeh, A. (2022). Phase Transformation Kinetics in Laser-Powder Bed Fused Fe-Cr-Ni-Al Maraging Stainless Steel. Available at SSRN: https://ssrn.com/abstract=4156053 or http://dx.doi.org/10.2139/ssrn.4156053
18. Ge, Hong-Guang & Jiao, Bao-Juan & Shuai, Qi & Liu, Ming-Yan & Chen, San-Ping & Hu, Rong-Zu & Gao, Sheng-Li. (2005). Determination of the Specific Heat Capacity of RE(Et2dtc)3(phen) by Microcalorimetry. Chinese Journal of Chemistry - CHINESE J CHEM. 23. 1495-1498. 10.1002/cjoc.200591495.
19. Demirtürk, Duygu & Ozturk, Hande & Güler, Murat. (2023). Using Calorimetry-Based Method for Measuring Specific Heat Capacity of Asphalt Concrete. 10.31462/icearc.2023.tra873.
20. Zhu, H., Lu, H., Zhang, Y., Xu, H., Brodský, J., Gablech, I., Feng, J., Yan, Q., & Neuzil, P. (2024). Concurrent determination of heat and capacity change of a sessile droplet using a single measurement. *Sensors and Actuators A: Physical*, *378*, 116042. https://doi.org/10.1016/j.sna.2024.116042
21. Wada, B. C., Baldwin, O. W. M., & Van Hecke, G. R. (2023). Heat–Cool: A Simpler Differential Scanning Calorimetry Approach for Measuring the Specific Heat Capacity of Liquid Materials. *Thermo*, *3*(4), 537-548. https://doi.org/10.3390/thermo3040032
22. Gabriel Glotz, Donald J. Knoechel, Philip Podmore, Heidrun Gruber-Woelfler, and C. Oliver Kappe. *Organic Process Research & Development* 2017 *21* (5), 763-770. DOI: 10.1021/acs.oprd.7b00092
23. Petrucci, R. H., Herring, F. G., Madura, J. D., & Bissonnette, C. (2023). *Petrucci's General Chemistry: Modern Principles and Applications, eBook*. Pearson Higher Ed. 1528 p. https://books.google.com.ua/books/about/Petrucci_s_General_Chemistry_Modern_Prin.html?id=m-nMEAAAQBAJ&redir_esc=y
24. Vlassak J. J. Calorimetry: An Old Tool Revisited at the Nano-Scale. 2022. https://mse.rpi.edu/seminars/2022/calorimetry-old-tool-revisited-nano-scale
25. Steinemann, F.L., Rütti, D.P., Moser, M. *et al.* Simultaneous determination of enthalpy of mixing and reaction using milli-scale continuous flow calorimetry. *J Flow Chem* 12, 389–396 (2022). https://doi.org/10.1007/s41981-022-00237-x
26. Bhatnagar, S., Comerford, A., Xu, Z., Polato, D. B., Banaeizadeh, A., & Ferraris, A. (2024). Chemical Reaction Neural Networks for fitting Accelerating Rate Calorimetry data. *Journal of Power Sources*, *628*, 2025, 235834. https://doi.org/10.1016/j.jpowsour.2024.235834

27. Pushp, M., Lonnermark, A., & Vikegard, P. (2023). Ageing tests closer to real service conditions using hyper-sensitive microcalorimetry, a case study on EPDM rubber. *Polymer Testing*, *120*, 107948. DOI: 10.1016/j.polymertesting.2023.107948.
28. Alexandra Navrotsky. New Developments in the Calorimetry of High-Temperature Materials. *Engineering*, 2019, 5(3): 366–371 https://doi.org/10.1016/j.eng.2019.03.003.
29. Sun Woong Baek, Kira E. Wyckoff, Daniel D. Robertson, Matevž Frajnkovič, Yucheng Zhou, Sarah H. Tolbert, Ram Seshadri, and Laurent Pilon. Operando Calorimetry Investigation of Particle Size Effects on Heat Generation in Wadsley–Roth $(W_{0.2}V_{0.8})_3O_7$-Based Electrodes. ACS Applied Energy Materials 2023 6 (3), 1355-1367. DOI: 10.1021/acsaem.2c03150
30. Cunningham, W. S., Lei, T., Howard, H. C., Rupert, T. J., & Gianola, D. S. (2025). Kinetics of Amorphous Defect Phases Measured Through Ultrafast Nanocalorimetry. *Physical Review Letters*, *131*(21), 216102. https://doi.org/10.1103/PhysRevLett.131.216102
31. Ma, S., Zhang, X., Zheng, G., Qian, M., & Geng, L.. A study on martensitic transformation behavior in shape memory alloys via a modulated differential scanning calorimetry technique. *Appl. Phys. Lett.* 125, 252202 (2024). https://doi.org/10.1063/5.0240749
32. Yuyan Zhang; Srinivas Tadigadapa. Thermal characterization of liquids and polymer thin films using a microcalorimeter. *Appl. Phys. Lett.* 86, 034101 (2005). https://doi.org/10.1063/1.1850186
33. Lalaso V. Mohite, Vinay A. Juvekar, and Jyoti Sahu. Industrial & Engineering Chemistry Research 2019 *58*(18), 7495-7510. DOI: 10.1021/acs.iecr.8b04792
34. Joanna Drzeżdżon, Dagmara Jacewicz, Alicja Sielicka, Lech Chmurzyński,.Characterization of polymers based on differential scanning calorimetry based techniques, TrAC Trends in Analytical Chemistry, Volume 110, 2019, Pages 51-56, https://doi.org/10.1016/j.trac.2018.10.037 .
35. Musuc, A. M., Doni, M., & Popa, V. T. (2022). Microcalorimetric Characterization of Polymer Composites Biodegradability. *Engineering Proceedings*, *19*(1), 34. https://doi.org/10.3390/ECP2022-12664
36. Malik A. Blackman, Meisha L. Shofner, and Camden A. Chatham. Investigating Fast Scanning Calorimetry and Differential Scanning Calorimetry as Screening Tools for Thermoset Polymer Material Compatibility with Laser-Based Powder Bed Fusion. ACS Applied Polymer Materials 2025 7(2), 719-728. DOI: 10.1021/acsapm.4c03052
37. Mattia Isola, Giovanna Colucci, Aleandro Diana, Agusti Sin, Alberto Tonani, Valter Maurino, Thermal properties and decomposition products of modified cotton fibers by TGA, DSC, and Py–GC/MS, Polymer Degradation and Stability, Volume 228, 2024, 110937, https://doi.org/10.1016/j.polymdegradstab.2024.110937 .
38. Ganeeva, Y.M., Okhotnikova, E.S., Yusupova, T.N. et al. Thermal characteristics of polymer modified bitumen according to simultaneous thermal analysis and microcalorimetry investigation. J Therm Anal Calorim (2025). https://doi.org/10.1007/s10973-025-14036-8.

39. Hu, H., Wu, J., & Zhang, M. (2024). Microcalorimetry Techniques for Studying Interactions at Solid–Liquid Interface: A Review. *Surfaces*, *7*(2), 265-282. https://doi.org/10.3390/surfaces7020018.
40. Guerrero-Fajardo, C. A., Giraldo, L., & Moreno-Piraján, J. C. (2020). Graphene Oxide: Study of Pore Size Distribution and Surface Chemistry Using Immersion Calorimetry. *Nanomaterials*, *10*(8), 1492. https://doi.org/10.3390/nano10081492.
41. Chaillot, D.; Folliard, V.; Miehé-Brendlé, J.; Auroux, A.; Dzene, L.; Bennici, S. Basic Properties of MgAl-Mixed Oxides in CO_2 Adsorption at High Temperature. Materials 2023, 16, 5698. https:// doi.org/10.3390/ma16165698.
42. Kevin Raheem, John Cassidy, Anthony Betts, Bernard Ryan, A simple microcalorimetry system to determine the adsorption behaviour of acids in large adhesive bond gaps using base-initiated solution polymerisation of ethyl-2-cyanoacrylate, International Journal of Adhesion and Adhesives, Volume 125, 2023, 103424, DOI: 10.1016/j.ijadhadh.2023.103424
43. Rixiang Huang, Boris L.T. Lau, Biomolecule–nanoparticle interactions: Elucidation of the thermodynamics by isothermal titration calorimetry, Biochimica et Biophysica Acta (BBA) - General Subjects, Volume 1860, Issue 5, 2016, Pages 945-956, https://doi.org/10.1016/j.bbagen.2016.01.027
44. Prozeller, D., Morsbach, S., & Landfester, K. (2019). Isothermal titration calorimetry as a complementary method for investigating nanoparticle–protein interactions. *Nanoscale*, *11*(41), 19265–19273. DOI: 10.1039/C9NR05790K
45. Rodler, A., Ueberbacher, R., Beyer, B., & Jungbauer, A. (2019). Calorimetry for studying the adsorption of proteins in hydrophobic interaction chromatography. Preparative Biochemistry & Biotechnology, 49(1), 1–20. https://doi.org/10.1080/10826068.2018.1487852
46. Abian, O., Vega, S., & Velazquez-Campoy, A. (2023). Biological Calorimetry: Old Friend, New Insights. *Biophysica*, *3*(1), 21-34. https://doi.org/10.3390/biophysica3010002
47. Kabiri, M., & Unsworth, L. D. (2014). Application of Isothermal Titration Calorimetry for Characterizing Thermodynamic Parameters of Biomolecular Interactions: Peptide Self-Assembly and Protein Adsorption Case Studies. *Biomacromolecules*, *15*(10), 3463–3473. DOI: 10.1021/bm5004515.
48. O'Brien, Ronan, and John E Ladbury, Isothermal titration calorimetry of biomolecules', in Stephen E Harding, and Babur Z Chowdhry (eds), *Protein-Ligand Interactions: hydrodynamics and calorimetry* (Oxford, 2000; online edn, Oxford Academic, 31 Oct. 2023), https://doi.org/10.1093/oso/9780199637492.003.0010, accessed 2 July 2025.
49. K. Joyce, S. Rahmani, Y. Rochev, Quasi-isothermal modulated DSC as a valuable characterisation method for soft tissue biomaterial crosslinking reactions, Bioactive Materials, Volume 5, Issue 2, 2020, Pages 428-434. https://doi.org/10.1016/j.bioactmat.2020.03.002.

50. Armen, J. M. (2022). BIOMATERIALS: FROM SCAFFOLD DESIGN TO IMPLANT OPTIMIZATION (Doctoral dissertation, Duquesne University). Retrieved from https://dsc.duq.edu/etd/2160.
51. De Klerk WPC (1996). *Thermal analysis of some propellants and explosives with DSC and TG/DTA* (ADA320678). Defense Technical Information Center. https://apps.dtic.mil/sti/tr/pdf/ADA320678.pdf.
52. https://standards.globalspec.com/std/14609978/stanag-4147.
53. De Klerk WPC (2015). Assessment of stability of propellants and safe lifetimes. Propellants, Explosives, Pyrotechnics 40(3):388-393. https://doi.org/10.1002/prep.201500040.
54. Turcotte, R.; Fouchard, R.C.; Turcotte, A.-M.; Jones, D.E.G. Thermal analysis of black powder.*J. Therm. Anal. Calorim.* 2003, *73*, 105–118. https://link.springer.com/article/10.1023/A:1025181424038
55. Liang, Xue & Zhao, Feng-Qi & Xing, Xiao-Ling & Xu, Si-Yu & Qing, Pei & Yin, Gao & Rong-Zu, Hu. (2010). Evaluation of Thermal Hazard of Composite Modified Double-base Propellant by Microcalorimetery Method. Chemical Research in Chinese Universities. 26(4) https://www.researchgate.net/publication/268437233_Evaluation_of_Thermal_Hazard_of_Composite_Modified_Double-base_Propellant_by_Microcalorimetery_Method
56. Svensson, L.G. & Paulsson, L.E. & Lindblom, Torbjörn. (1990). A Microcalorimetric Study of Temperature and Stabilizer Effects on the Heat Generation in Gun Propellants. 4th Gun Propellant Conference. Mulvala, Australia, 1990. https://www.researchgate.net/publication/291946415_A_Microcalorimetric_Study_of_Temperature_and_Stabilizer_Effects_on_the_Heat_Generation_in_Gun_Propellants.
57. Svensson, L.G. (2009). Ampoule microcalorimetry for stability and Compatibility Testing of Explosives and materials. Engineering, Materials Science, Chemistry.
58. Folly, P. (2004). Thermal Stability of Explosives. *CHIMIA*, *58*(6), 394–400. https://ojs.chimia.ch/chimia/article/download/3863/3153/13838
59. Mirjana Dimić, Bojana Fidanovski, Ljiljana Jelisavac, Vesna Rodić. Analysis of the of Propellants-Polymers Compatibility by Different Test Methods. Scientific Technical Review, 2017, Vol. 67, No.2, pp.13-19. https://pdfs.semanticscholar.org/5149/7931eb4e8e06e5518f4764b7298550e6b345.pdf
60. Vogelsanger, Beat. (2004). Chemical Stability, Compatibility and Shelf Life of Explosives. CHIMIA International Journal for Chemistry. 58. 401-408. 10.2533/000942904777677740.
61. La Haye, Ellen & Klerk, W. & Miszczak, M. & Szymanowski, J. (2003). Compatibility testing of energetic materials at TNO-PML and MIAT. Journal of Thermal Analysis and Calorimetry – J THERM ANAL CALORIM. 72. 931-942. 10.1023/A:1025034719070.
62. Roduit, B. & Borgeat, Ch & Berger, B. & Folly, Patrick & Andres, H. & Schädeli, U. & Vogelsanger, B. (2006). Up-scaling of DSC data of high energetic materials – Simulation of cook-off experiments. Journal of Thermal Analysis and Calorimetry. 85. 195–202. DOI:10.1007/s10973-005-7388-y

63. Stanković, M., Antić, G., Blagojević, M. *et al.* Microcalorimetric Compatibility Testing of the Constituents of Combustible Materials and Casting Composite Explosives. *Journal of Thermal Analysis and Calorimetry* 52, 581–585 (1998). https://doi.org/10.1023/A:1010127924628
64. Jimmie C. Oxley. Chapter 8 - The Thermal Stability of Explosives, Handbook of Thermal Analysis and Calorimetry, Volume 2, 2003, Pages 349-369. https://doi.org/10.1016/S1573-4374(03)80012-2.
65. Fidanovski, Bojana & Simic, Danica & Dimić, Mirjana & Terzić, Slavica. (2020). Differential scanning calorimetry and vacuum stability test as methods to determine explosives compatibility. Scientific Technical Review. 70. 35-40. 10.5937/str2003035F.
66. Maule, I., Razzetti, G., Restelli, A., Palmieri, A., Colombo, C., & Ballini, R. (2021). Organic Process Research & Development, *25*(4), 781–788. https://doi.org/10.1021/acs.oprd.0c00433
67. Sanchirico, R., & Di Sarli, V. (2023). An Alternative Approach for Predicting the Shelf Life of Energetic Materials. *Fire*, *6*(9), 333. https://doi.org/10.3390/fire6090333.
68. Stolarczyk, A., & Jarosz, T. (2022). Thermal Properties of Energetic Materials–What Are the Sources of Discrepancies? *Fire*, *5*(6), 206. https://doi.org/10.3390/fire5060206
69. Huestis, P. L., Stull, J. A., Lichthardt, J. P., Wasiolek, M. A., Montano-Martinez, L., & Manner, W. V. (2022). Effects of Low-Level Gamma Radiation on Common Nitroaromatic, Nitramine, and Nitrate Ester Explosives. *ACS Omega*, *7*(3), 2842–2849. https://doi.org/10.1021/acsomega.1c05703.
70. Tarchoun, A. F., Trache, D., Abdelaziz, A., Harrat, A., Boukecha, W. O., Hamouche, M. A., Boukeciat, H., & Dourari, M. (2022). Elaboration, Characterization and Thermal Decomposition Kinetics of New Nanoenergetic Composite Based on Hydrazine 3-Nitro-1,2,4-triazol-5-one and Nanostructured Cellulose Nitrate. *Molecules*, *27*(20), 6945. https://doi.org/10.3390/molecules27206945
71. Zuo, Chun-jie & Zhang, Chao-yang. (2024). Standardizing differential scanning calorimetry (DSC) thermal decomposition temperatures at various heating rates of an energetic material as a threshold one. Energetic Materials Frontiers. 10.1016/j.enmf.2024.06.006.
72. Roseres, C.-A., Courty, L., Gillard, P., Boulnois, C., & Le Renard, C. (2019, June). *Reactivity of pyrotechnic compositions: Influential parameters study*. 44th International Pyrotechnics Society Seminar - Europyro 2019, Tours, France. ⟨hal-02172931⟩.
73. Cheng, J., Yan, N., Ye, Y. *et al.* Stability of pyrotechnic composition in flame detonator exposed to severe thermal stimulus. *Chem. Res. Chin. Univ.* 31, 814–819 (2015). https://doi.org/10.1007/s40242-015-5083-5
74. Phanindra, K. H., Sivapirakasham, S. P., Balasubramanian, K. R., & Surianarayanan, M. (2019). Thermal and Impact Sensitivity of Pyrotechnic Compositions Containing the Potassium Perchlorate, Aluminium and Graphite. Materials Science Forum, 969, 146–151. https://doi.org/10.4028/www.scientific.net/msf.969.146

75. Sivapirakasam, S. P., Surianarayanan, M., & Vijayaraghavan, R. (2006). Thermal Characterization And Kinetic Modeling Of A Pyrotechnic Flash Composition Under Adiabatic Conditions. *Journal of Pyrotechnics*, *(23)*, 61–67. https://www.researchgate.net/publication/249645232
76. Sivapirakasam, S. P., Surianarayanan, M., & Vijayaraghavan, R. (2010). Thermal Characterization of Pyrotechnic Flash Compositions. *Science and Technology of Energetic Materials*, *71*(1), 11–16. https://www.researchgate.net/publication/286176243
77. Pakkirisamy, S. V., Mahadevan, S., Paramashivan, S. S., & Baran, M. A. (2014). Water induced thermal decomposition of pyrotechnic mixtures. Thermokinetics and explosion pathway. *Journal of Loss Prevention in the Process Industries*, *30*, 275-281. https://doi.org/10.1016/j.jlp.2014.03.005 https://doi.org/10.1016/j.jlp.2014.03.005.
78. Li, Q., Yao, P., Wang, L., & Zhang, W. (1995). The application of microcalorimetry in the compatibility test for pyrotechnics. *Thermochimica Acta*, *253*, 213–220. https://doi.org/10.1016/0040-6031(94)02097-8
79. Xia, H. (2017). Thermal Analysis and Thermal Hazard Analysis of Ionic Liquids (Version 1). University of Notre Dame. https://doi.org/10.7274/zg64th86c21.
80. Rongzu, H., Xuelin, C., Shijin, C. *et al.* A new method of determining the thermal conductivities of energetic materials by microcalorimeter. *Journal of Thermal Analysis* 42, 505–520 (1994). https://doi.org/10.1007/BF02548532.
81. Wim de Klerk, Niels van der Meer, Reinier Eerligh. Microcalorimetric study applied to the comparison of compatibility tests (VST and IST) of polymers and propellants. Thermochimica Acta. Volumes 269–270, 20 December 1995, Pages 231-243. https://doi.org/10.1016/0040-6031(95)02363-1.
82. Svensson I. Using isothermal microcalorimetry for the prediction and testing of long-term properties of materials and products. Journal of Thermal Analysis and Calorimetry. Volume 49: Issue 2, 1017–1023. https://doi.org/10.1007/bf01996789
83. Guillaume, Pierre & Rat, Mauricette & Pantel, Gabriele & Wilker, Stephan. (2001). Heat Flow Calorimetry of Propellants - Effects of Sample Preparation and Measuring Conditions. Propellants Explosives Pyrotechnics - PROPELLANT EXPLOS PYROTECH. 26. 51-57. 10.1002/1521-4087(200104)26:23.0.CO;2-H.
84. Brown, S. D., Charsley, E. L., Goodall, S. J., & Laye, P. G. (2003). Studies on the ageing of a magnesium–potassium nitrate pyrotechnic composition using isothermal heat flow calorimetry and thermal analysis techniques. Thermochimica Acta, 401(1), 53–61. https://doi.org/10.1016/S0040-6031(03)00055-8.
85. Croce, M.P., Bracken, D.S., Likes, R.N., Rudy, C.R., Santi, P.A. (2024). Principles of Calorimetric Assay. In: Geist, W.H., Santi, P.A., Swinhoe, M.T. (eds) Nondestructive Assay of Nuclear Materials for Safeguards and Security. Springer, Cham. https://doi.org/10.1007/978-3-031-58277-6_23.
86. Amharrak, H., Reynard-Carette, C., Lyoussi, A., Carette, M., Brun, J., De Vita, C., Fourmentel, D., Villard, J.-F., & Guimbal, P. (2014). Monte Carlo Simulation Study of a

Differential Calorimeter Measuring the Nuclear Heating in Material Testing Reactors. *EPJ Web of Conferences, 106*, 05005. https://doi.org/10.1051/epjconf/201610605005.

87. Zhang, J., Ma, T., Zhang, L., Peng, X., Qu, G., Jin, S., Sun, Y., Han, J., & Zhao, G. (2025). The design and calibration of a new differential calorimetric dedicated to nuclear heating measurements. *Annals of Nuclear Energy, 219*, 111485. https://doi.org/10.1016/j.anucene.2025.111485

Лисько В.В.[1,2] (https://orcid.org/0000-0001-7994-6795),
Микитюк О.Ю.[1,3] (https://orcid.org/0000-0001-9365-4836)

[1]Інститут термоелектрики НАН та МОН України,
вул. Науки, 1, Чернівці, 58029, Україна;
[2]Чернівецький національний університет імені Юрія Федьковича,
вул. Коцюбинського 2,Чернівці, 58012, Україна;
[3]Буковинський державний медичний університет,
Театральна площа, 2, Чернівці, 58002, Україна

Застосування мікрокалориметрії у матеріалознавстві

В цій оглядовій статті розглянуто практичне використання калориметричних методів у матеріалознавстві, зокрема для вивчення фазових переходів, визначення теплоємності та теплових властивостей, дослідження дефектів і структурних змін, дослідження полімерів, біоматеріалів, поверхневих явищ та ін. Висвітлено застосування мікрокалориметрії для контролю вибухових речовин та піротехнічних засобів.

Ключові слова: мікрокалориметрія, матеріалознавство, фазові переходи, полімери, біоматеріали, вибухові речовини.