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RESEARCH IN THE LABORATORY OF THERMOMECHANICS, APPLIED THERMAL ENERGY, AND NANOTECHNOLOGIES

RAZISKAVE V LABORATORIJU ZA TERMOMEHANIKO, TERMOENERGETIKO IN NANOTEHNOLOGIJE

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Abstract

This article presents the research work in the Laboratory of Thermomechanics, Applied Thermal Energy, and Nanotechnologies at the Faculty of Energy Technology of the University of Maribor. The most significant work that has been done is presented.

Povzetek

Članek predstavlja raziskovalno delo v laboratoriju za termomehaniko, termoenergetiko in nanotehnologije. V članku so predstavljene glavne dejavnosti laboratorija in nekaj pomembnih raziskovalnih dosežkov.

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1 THERMAL ANALYSIS AND CALORIMETRY

Among other activities, the Laboratory for Thermomechanics, Applied Thermal Energy Technologies, and Nanotechnologies deals with thermal analysis and calorimetry.

According to *[1],* Thermal Analysis (TA) is the analysis of a change in a sample property (e.g. thermodynamic properties (heat, temperature, mass, enthalpy, etc.), material properties (hardness, susceptibility, etc.), chemical composition or structure) that is related to a specific, imposed temperature alteration. Calorimetry is the measurement of heat.

For the implementation of the above-mentioned analysis, the laboratory has relevant equipment, including a Mettler Toledo TGA/DSC 3+ with the STAR^{e} system, a Pfeiffer Vacuum Thermostar™ gas analysis system, a Thermo scientific TGA-IR, a Nicolet iS50 FTIR for thermal analysis, and aMettler Toledo EasyMax® 102 for heat flow calorimetry (see Figures 1 and 2).

Figure 1: *Mettler Toledo TGA/DSC 3+ (left), Pfeiffer Vacuum Thermostar™ (right)*

Figure 2: *Thermo scientific TGA-IR and Nicolet iS50 FTIR (left), Mettler Toledo EasyMax 102 (right)*

The Mettler Toledo TGA/DSC 3+ device enables several thermal analysis techniques:

- DTA (Differential Thermal Analysis), in which the temperature difference between the sample and an inert reference substance is measured while being subjected to a temperature alteration, *[1, 2];*
- DSC (Differential Scanning Calorimetry), in which the heat flow in and out of a sample and a reference substance is measured as a function of temperature. With the DSC, exothermic and endothermic effects can be detected, specific heat capacities can be measured, etc., *[2];*
- TGA (Thermogravimetric Analysis), in which the change in the sample mass as a function of temperature is analysed, *[1].* With the TGA, stepwise changes in mass, temperatures that characterize a step in the mass loss, etc, can also be determined, *[2].*

An example of the TGA and DSC curves of the decomposition of Calcium Oxalate Monohydrate ($CaC_2O_4.H_2O$) is shown in Figure 3. Both curves are functions of temperature. Changes in sample mass (in our case mass loss) are shown on the blue curve (TGA curve) as a percentage of the initial sample mass. The violet curve (DSC curve) shows the amount of energy absorbed by the sample (in milliwatts); these are endothermic effects.

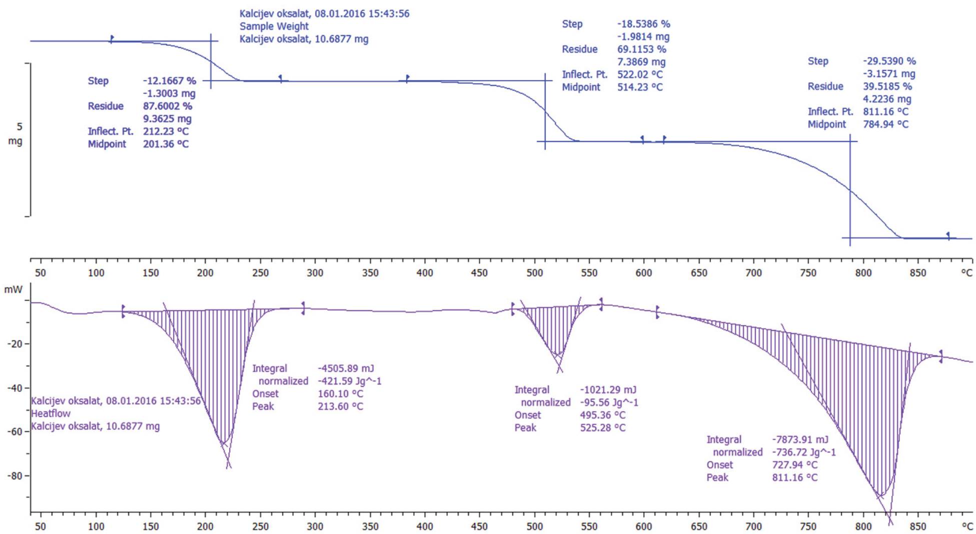

Figure 3: *TGA (above) and DSC (below) curves of the decomposition of Calcium Oxalate Monohydrate*

As can be seen from Figure 3, the TGA is not an identification technique. It cannot identify what the gaseous products that evolved during a TGA measurement are. It can be asserted that the gaseous products with the combination of TGA/DSC with a MS (mass spectrometer) or FTIR (Fourier transform infrared spectrometer). The technique is called EGA (Evolved Gas Analysis), which is the analysis of gases that are evolved from a sample undergoing thermal analysis, *[3].*

However, great care must be taken with the interpretation of results. Time is the parameter common to all instruments (TGA/DSC, MS and FTIR). The data must be correlated mutually.

Mass spectrometry is an extremely sensitive analytical technique for the detection and identification of gaseous substances. The method quantifies atoms or molecules and provides chemical and structural information on the traces of gaseous substances analysed, *[3].*

Fourier transform infrared spectroscopy is a technique in which IR radiation is passed through a sample. It can identify unknown substances, determine the quality of a sample or the amount of components in a substance. FTIR is less sensitive than MS, *[4].*

The Mettler Toledo EasyMax® is a reaction calorimeter for heat flow calorimetry. The heat flow across the reactor wall and quantification of this in relation to the other energy flows inside the reactor is what heat flow calorimetry measures, *[5]*. This technique allows us to measure heat whilst the process temperature is controlled. With the heat flow calorimetry, the heat transfer coefficient, reaction enthalpy, the amount of energy absorbed or released by a chemical reaction, etc. can be determined.

2 MEASUREMENTS OF EFFICIENCIES IN BOILERS, HEAT EXCHANGERS, AND HEAT PUMPS

For measurements of efficiency in furnaces, solar collectors, and heat pumps, an entire measuring line must be set up. The heat produced during the measurement is transmitted to a central storage room where heat is then used for heating. Measuring equipment can be used to measure efficiency (energy and exergy efficiency), temperature, and pressure in power systems (see Figures 4 and 5). For the determination of temperature fields, thermovision equipment mounted on drones is also used (Figure 6).

With the measuring system, the following can be measured:

- energy efficiency of boilers, heat pumps, and solar systems,
- exergy efficiency of boilers, heat pumps, and solar systems,
- temperatures in all important points,
- emissions in the case if we test boilers with fossil fuels and biomass.

***Figure 4**: Some details for the thermal part of the laboratory*

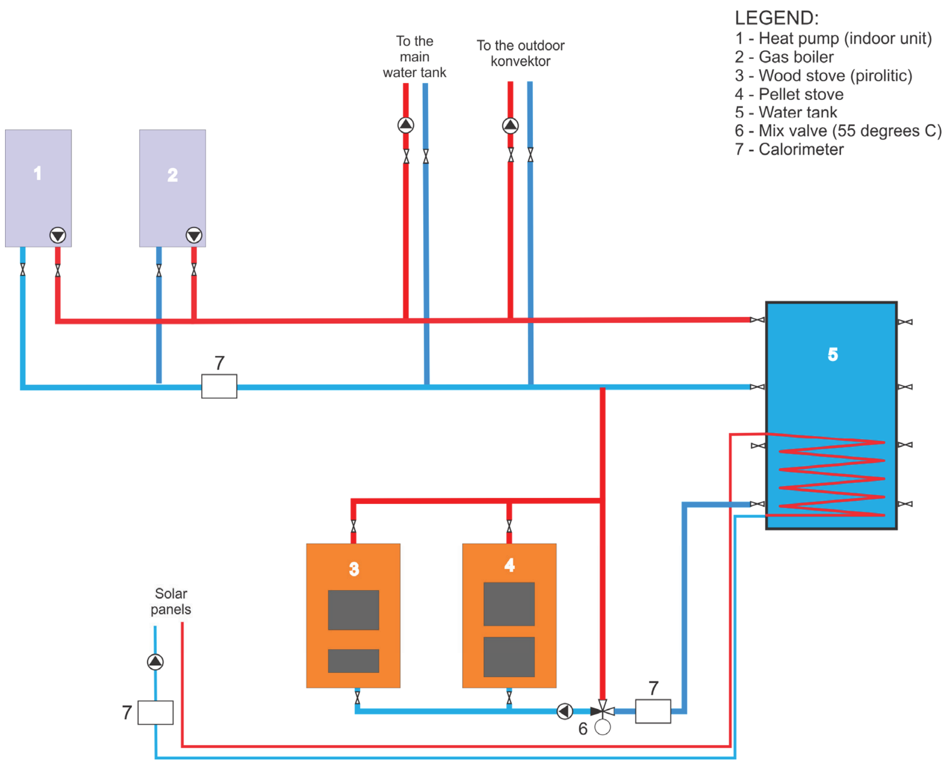

***Figure 5:** Measuring principle line for thermal part of the laboratory*

***Figure 6**: Thermovisional inspections using drones*

3 HYDROGEN TECHNOLOGIES

At the laboratory, we are very interested in the development of methanol technologies and hydrogen technologies. The development in this area is carried out in two segments:

1. Hydrogen production. In this field we are developing new hydrogen production systems. We are mainly engaged in the development of hydrogen production from water in connection with a thermoelectric, nuclear power plant or solar thermal power plant. Hydrogen can be obtained from water by means of electrolysis, thermochemical processes, photochemical processes, biochemical processes, and biological processes.

2. Use of hydrogen and methanol. To this end, we are involved in the development and application of fuel cells and hydrogen, Methanol for fuel cell transport will be of vital importance in the future. Normally, fuel cells are divided into groups according to the type of the electrolyte they use. Alkaline fuel cells (AFC) operate optimally within the temperature range of 1200 °C–2500 °C, with concentrated KOH utilized as the electrolyte. A proton exchange membrane fuel cell (PEMFC) uses a very thin polymer membrane as the electrolyte. Its working temperature ranges approximately from 60 °C–80 °C and it currently represents the most promising fuel cell for installation in cars, trains etc. A solid oxide fuel cell (SOFC) uses non-porous metal oxide as the electrolyte. These cells operate at higher temperatures, 600 °C–1000 °C. Also known are phosphoric acid fuel cells (PAFC), and molten carbonate fuel cells (MCFC), but these are not used

very often. At present, all the major automotive factories have their own system of powering vehicles with fuel cells. Due to significant overpopulation and being highly export-oriented, two Japanese factories (Honda and Toyota) have become highly developed in this field. However, other companies and automotive groups are not far behind. On the market today are some transport vehicles, such as the Toyota Mirai and Hyundai IX 35 Fuel cell (Figure 7).

Figure 7: *Hydrogen fuel station with Toyota Mirai car*

4 THERMOMECHANICS (MACRO-, MICRO-, AND NANO-LEVELS)

One of the great scientific and technical advancements of the end of 20^{th} and at start of the 21^{st} century was the creation of nanomaterials and nanomechanics. The area that covers all important problems from that field is called, in the broadest sense, “mechanics”. Regarding the cross-sectional diameter, mechanics can be divided into subdisciplines: [6]

macromechanics 10^{-4}-10^{-5} m,

mesomechanics 10^{-5}-10^{-7} m,

micromechanics 10^{-7}-10^{-8} m,

nanomechanics: 10^{-8}-10^{-9} m.

Since the atomic level (interatomic distance in a crystal lattice) has an order of one to several Å (10^{-10} m), the nanolevel is restricted to 10^{-9} m. Accurate data for thermodynamic and transport properties of gases and liquids is crucial for the achievement of many technological goals in thermal and fluid engineering systems. It is estimated that about 50 million pure substances are known today, but with only some 20,000 substances being recorded in journals and manuals. There are approximately 30 thermo-mechanical properties for each pure substance, which are essential to engineering practice (about 12 of which are dependent on pressure and temperature). If the transport properties of every pure substance were measured at ten different temperatures and ten different pressures, then 1,200 measurements would be needed for each pure substance. Over 100 trillion years of work would be needed to carry out measurement of all properties for every pure substance and known mixtures. Thus, measurements alone are unfeasible and, therefore, analytical methods to determine transport properties are essential. It is possible to determine thermophysical properties by conventional thermodynamics by experimental work or the use of statistical thermodynamics. For the calculation of thermodynamic and transport properties, conventional and statistical thermomechanical methods can be used. The calculated thermophysical properties are used in the further calculation of fluid mechanics, the mechanics of materials, and software to calculate the real behaviour of energy devices.

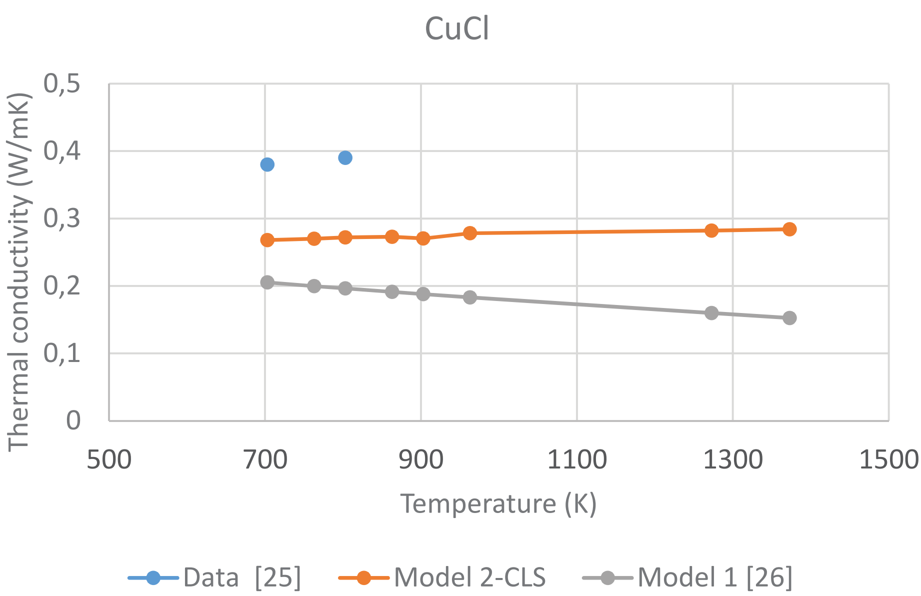

Figure 8: *Thermal conductivity of molten CuCl, [7]*

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Nomenclature

TA	Thermal Analysis
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
FTIR	Fourier Transform InfraRed
DTA	Differential Thermal Analysis
DSC	Differential Scanning Calorimetry
MS	Mass Spectrometer/mass spectrometry
EGA	Evolved Gas Analysis