PROBLEMY MECHATRONIKI
UZBROJENIE, LOTNICTWO, INŻYNIERIA BEZPIECZEŃSTWA

ISSN 2081-5891 **12, 2 (44), 2021, 9-38**

PROBLEMS OF MECHATRONICS
ARMAMENT, AVIATION, SAFETY ENGINEERING

Thermal Properties of Solids – Theoretical Basis, Research Methods and Selected Results of Proprietary Research

(*The paper was commissioned by the Editors Office of the quarterly*)

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Received by the editorial staff on 16 March 2021
The reviewed and verified version was received on 28 May 2021

DOI 10.5604/01.3001.0014.9331

Abstract. This paper refers to an inaugural lecture prepared by the author for the inauguration of the New Academic Year 2020/2021 at the Faculty of Mechatronics, Armament and Aerospace of Military University of Technology (MUT) in Warsaw (Poland) on 2 October 2020. It presents the origins of research into thermal properties of solids since the mid-1970s by the employees of the thermodynamic research unit at the Department of Aerodynamics and Thermodynamics, followed by the basic modalities of heat transfer, theoretical foundations of thermal expansion, specific heat, thermal conductivity and thermal diffusivity of solids. The measuring apparatus created as a result of proprietary research studies and purchased from market-leading manufacturers is shown with a selection of results from the research into the thermal properties of solids, which are largely the outcome of the application our own research procedures.

Keywords: heat transfer, thermophysical parameters, test procedures, test stands

1. INTRODUCTION

The Department of Aerodynamics and Thermodynamics (DAT) of the Military University of Technology (MUT, Warsaw, Poland)) is a research and teaching unit recognised by both national and international scientific centres. The year in which the Department was formally established under the direction of Col. Stefan Wiśniewski, Prof. PhD Eng., and the original name "The Department of Thermodynamics and Combustion" (DTC) is assumed to be 1967. The Department was established by divesting it from the Department of Aircraft and Missile Engines (DAME), which was headed since 1954 by Brigadier General Ryszard Szymanik, Prof. PhD Eng., into an organisation the personnel of which had been tasked with teaching and research in the field of thermodynamical engineering within the DAME. In the years 1982-1998 the head of DAC was Col. Janusz Terpiłowski, Prof. PhD. Eng. Note, however, that since 1987 the name of the Department was changed several times in connection with its restructuring. Successively, it was the Department of Thermodynamics (1987-1993), the Department of Aircraft Propulsion and Thermodynamics (1993-2004), and finally, the Department of Aerodynamics and Thermodynamics (2004 to present). The succession of the Department directors included Col. Tadeusz Opara, Prof. of MUT PhD. Eng., Col. Stanisław Wrzesień, Prof. of MUT PhD. Eng., and, continuously from 2005 to 2020, Col. Piotr Koniorczyk, Prof. PhD. Eng.

The basic direction of research at the DAT Thermodynamics Team is experimental research into thermophysical properties of solids, in particular, research into temperature characteristics of thermal diffusivity, thermal conductivity, specific heat and thermal expansion. Numerical simulations and experimental studies of both heat transfer and heat and mass transfer are carried out in parallel. These include the complex issues of conduction coupled with radiative heat transfer in radiation emitting, absorbing and scattering media, heat and mass transfer in porous structures and heat transfer in biological structures. Important directions of research include analytical and numerical calculations of the thermal conductivity reduction effects on thin samples in optically active media, estimation of thermophysical parameters based on the solution of the inverse heat conduction problem, and numerical modelling of phase transitions of the first kind in phase change materials. The Thermodynamic Team also studies heat transfer in aerospace structures. This area was covered by Professor Stefan Wiśniewski's unique monograph titled "Thermal loads of turbine engines" (in Polish) published in 1974 by the Polish Publishing House "Wydawnictwo Komunikacji i Łączności" in Warsaw.

2. GENESIS OF THE RESEARCH

The Thermodynamics Team of the MUT Department of Aerodynamics and Thermodynamics, which the author has been a member of for nearly 40 years, is unique in Poland. Among its 6 employees there are 4 titular professors, who acquired their successive scientific degrees at MUT. All of them are primarily involved in research into thermal properties and are nationally and internationally recognised experts in this field. The study of thermophysical parameters is fundamental and the knowledge of it is essential in the process of design, manufacturing and engineering of components and numerical modelling of complex thermomechanical problems. For example, the resultant elongation of approximately 8 mm of a K-15 turbine blade airfoil, rotating at 15,600 rpm and exposed to exhaust gases of about 1,100 K, is affected 80% by thermal effects and only 20% by mechanical effects (Fig. 1). Measurement of the in-flight temperature on the leading edge of a wing of the TS-11 Iskra aircraft (Fig. 2), or of the temperature of a human tooth (Fig. 3) is necessary in the process of validating theoretical models of complex heat and mass transfer, but for this modelling, thermal characteristics of materials are necessary, which include specific heat, thermal conductivity, thermal diffusivity and thermal expansion.

$$\varepsilon_x = \frac{1}{\mathrm{E}}\left[\sigma_x - \nu\left(\sigma_y + \sigma_z\right)\right] + \alpha(T - T_0)$$

~ 20% ~ 80%

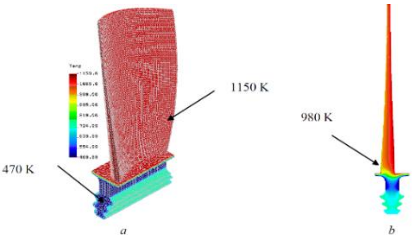


Fig. 1. Example result of numerical modelling of total elongation of a K-15 engine turbine blade under thermal loads [1]

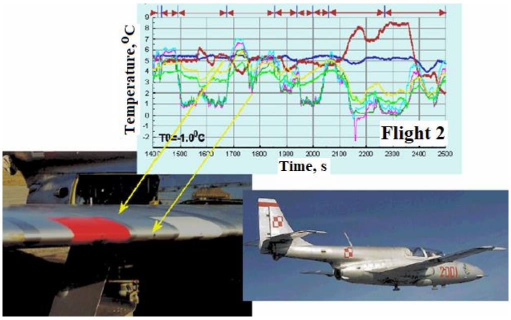


Fig. 2. Experimental results of in-flight temperature measurement of the leading edge of a wing of the TS-11 Iskra aircraft [2]

Thanks to the openness of the MUT's Management to cooperation with national research centres and industry, several studies of the MUT's proprietary test stands have been developed. These include Prof. Koniorczyk's (1987) test rig, built from scratch for testing of insulating materials at up to 700°C based on the Poensgen plate apparatus concept with a thermal shielding plate (Fig. 4).

Fig. 3. Setup of a human tooth for temperature measurement with thermocouples [3-5]

Fig. 4. Overview of the test stand for testing thermal conductivity of insulating materials from room temperature (RT) to 700°C, which was developed and built by Professor P. Koniorczyk at DAT in 1987 [6, 7]

The fabrication of dedicated heaters and coolers with a diameter of 200 mm is the result of cooperation with the National Centre for Nuclear Research in Świerk (Poland). The test stand for testing the specific heat of engine oils, shown in Fig. 5, is a proprietary work of Marek Preiskorn, PhD (1993). Noteworthy is the test stand for thermal diffusivity pulse testing of solids (Fig. 6), designed by Professor J. Terpiłowski in 1975. The steel vacuum furnace was made at the National Centre for Nuclear Research in Świerk, and the pulsed neodymium laser is a pioneering device in Poland, developed at the MUT Institute of Optoelectronics by the team headed by Professor Z. Puzewicz.

Fig. 5. Engine oil specific heat test stand (design and construction by M. Preiskorn, PhD Eng., 1993)

Fig. 6. Test stand for thermal diffusivity pulse testing of solids, designed by Professor J. Terpiłowski in 1975, and partly engineered at the National Centre for Nuclear Research in Świerk

The diffusometer test stand was one of the first of its kind in the world, but the scientific success of its implementation laid not so much in reproducing the state-of-the-art solutions at the time, but in developing them at the same time. Developed by Professor Janusz Terpiłowski, the method of measurement [8] originally removed the basic disadvantages of Parker’s original method [9]. A creative approach and development of new research methods by application of theoretical models of heat transfer phenomena or modelling of physical phenomena in a wider scope, is a distinctive feature of the work of the MUT Thermodynamics Team. The interference dilatometer with a flat-convex lens for testing the thermal expansion of solids shown in Fig. 7 was developed in 1985 (Polish Patent No. 260749, in 1988).

Thanks to the concept introduced by Professor A. Panas to use a plane-parallel spherical system for the detection of changes of length, it was possible to directly measure thermal expansion at a record-high temperature for this type of testing device.

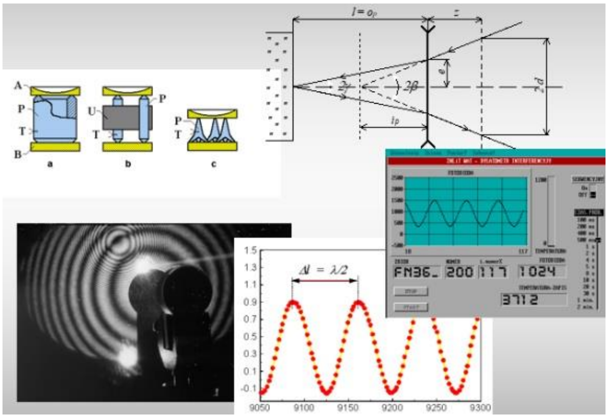


Fig. 7. Interference method thermal expansion test stand by A. Panas (1985) [10]

The interference dilatometer system was also the proving ground for the development of virtual instruments. The first controllers of this type, designed by J. Jóźwiak, Eng. and Professor A. Panas, were a result of the application of a combination of proprietary assembler programming routines and a basic language [3]. The team from the-then Department of Thermodynamics and Combustion Theory was one of the first in Poland to introduce working in LabWindows, TestPoint and later, LabVIEW virtual instrument programming packages. Another achievement of Prof. Panas (2002) is the development of a method for measuring the thermal diffusivity of solids using the step function method with the application of two fluids, together with the determination of metrological conditions. This method is an evolution of the classical Kondratiev method, and Fig. 8 shows the test stand for measurement of the thermal diffusivity by application of the developed procedures. In a similar way, the classical Ångström method has been modified to enable examination of phase transitions.

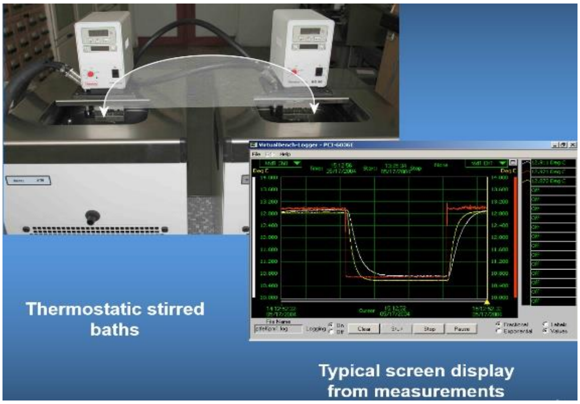


Fig. 8. Test stand for thermal diffusivity of solids by step function in two fluids under conditions of systematic heat exchange, by A. Panas (2002) [11]

The test stand originally used in the development phase of the method is shown in Fig. 9 (2005).

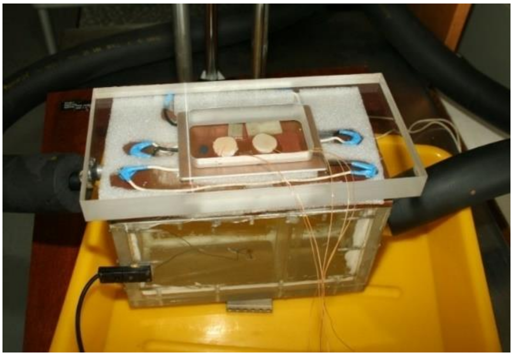


Fig. 9. Thermal diffusion test stand for the modified Angström method, by A. Panas (2005)

Figures 10 and 11 show the test stands for simultaneous identification of thermophysical parameters in solids by continuous heating (Professor P. Koniorczyk, 2010 – Fig. 10) and respectively, by inverse coefficient method with the use of an additional Kanthal wire heater arranged in a semi-circular form (Professor P. Zmywaczyk, 2006 – Fig. 11).

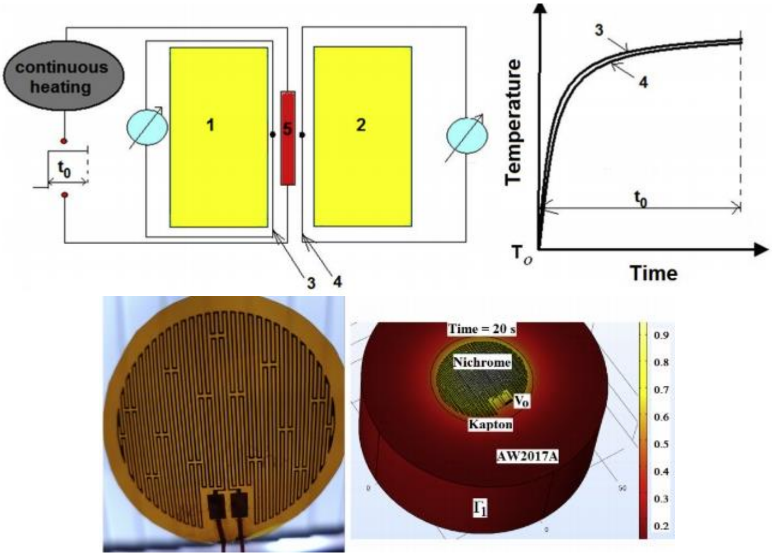


Fig. 10. Test stand for simultaneous identification of thermophysical parameters of solids by continuous heating, by P. Koniorczyk (2010) [12,13]

The test stands shown in Figs. 10 and 11 were developed and manufactured at the Department of Aerodynamics and Thermodynamics, MUT Institute of Aerospace Technology.

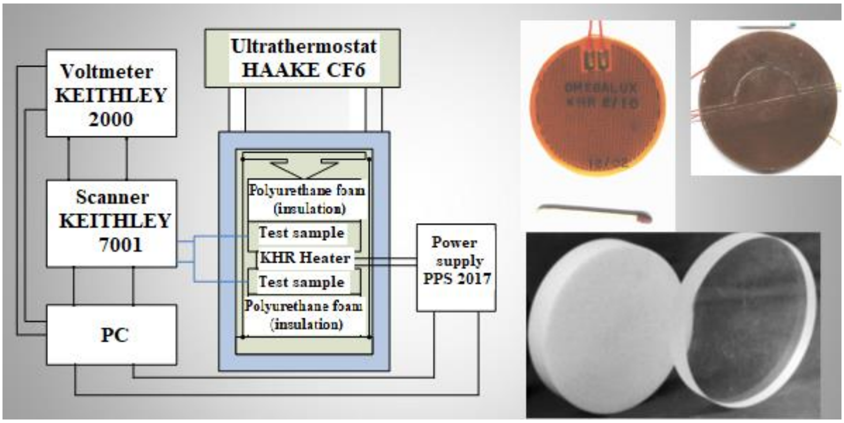


Fig. 11. Test stand for simultaneous identification of thermophysical parameters of solids by inverse coefficient method, J. Zmywaczyk (2006) [14]

3. BASIC HEAT TRANSFER MODALITIES

Heat transfer may occur by conduction, convection or radiation (Table 1). A precondition is the presence of a temperature difference. Fourier's law assumes that the heat flux density is proportional to the temperature gradient and that the process occurs at an infinitely high rate.

Table 1. Basic heat transfer modalities

Heat conduction	**Convection**	**Thermal radiation** (0.1 - 100) µm
Fourier's law (1822) $\dot{q}(r,t) = -\lambda \nabla T(r,t)$	Newton's law (1701) $\dot{q} = h(T - T_f)$	Stefan (1979) - Boltzmann (1884) law $\dot{q} = \sigma T^4$
Cattaneo (1958) - Vernotte (1961) $\dot{q}(r, t+\tau_q) = -\lambda \nabla T(r,t)$	$h = f$(geometry, laminar/turbulent flow, thermophysical parameters,...)	$\sigma = \frac{2\pi^5 k_B^4}{15 c^2 h^3} = 5{,}67 \cdot 10^{-8} \frac{\mathrm{W}}{\mathrm{m^2 K^4}}$
Tzou (1995, DPL model) $\dot{q}(r, t+\tau_q) = -\lambda \nabla T(r, t+\tau_T)$		

In the case of interaction of short laser pulses of 10^{-14} to 10^{-8} s with good heat conductors, the relaxation time of the heat flux density should be taken into account as $\tau_q = 10^{-12} - 10^{-8}$ s, and in the case of heat conduction through complex biological structures, which are multiscale three dimensional structures, the temperature gradient relaxation time should also be taken into account as τ_T.

This is another modification of Fourier's law proposed in 1995 by Tzou [15], called the dual-phase-lag (DPL) model. The mechanism of heat conduction in fluids (gases and liquids) is based on the transfer of kinetic energy to successive particles through elastic collisions, while in solids, heat conduction occurs mainly through phonons (in insulators) and electrons (in metals and metal alloys). Convection is the ascent of fluid macroscopic parts of higher temperature and the descent of fluid parts of a lower temperature, which replace the former parts. The density of the heat flux transferred is governed by Newton's law, which states that it is proportional to the difference in temperature of the solid T and the fluid $T_{f.}$ Thermal radiation may occur at a distance and involves successive processes of emission and absorption of EM waves, conventionally assuming a spectral range of radiation to be between 0.1 and 100 μm, which includes the ultraviolet, visible and infrared bands. The radiation flux density over the entire wavelength range is proportional to the 4th power of the absolute body temperature.

4. THERMOPHYSICAL PARAMETERS

4.1. Thermal expansion

Thermal expansion is the result of temperature changes in the mean equilibrium distance R_0 between adjacent atoms. The forces between the adjacent atoms at distance r from each other are of the nature of forces of attraction with an amplitude proportional to $1/r^7$ when $r > R_0$ and forces of repulsion varying as $1/r^{13}$ when $0 < r < R_0$ (Fig. 12a). In solids, as the temperature increases, the amplitude of vibration of the atoms relative to a specific equilibrium position increases. The resultant Morse curve, which takes into account the superposition of the repulsion (E_2) and attraction (E_1) potential energy curves, is asymmetric (E_3). Thus, the average equilibrium distance between adjacent atoms (the red dashed line in Fig. 12b) increases with increasing temperature. The result of an increase in body temperature is therefore an increase in the volume of that body.

The measure of thermal expansion of bodies is relative elongation ε, which is the ratio of the increase in length ΔL to initial length L_0

$$\varepsilon(T) = \frac{L(T) - L(T_0)}{L(T_0)} \tag{1}$$

The temperature derivative of the relative elongation of a body in a given direction is the coefficient of linear thermal expansion α^* (CLTE). Coefficient α^* is determined experimentally and its numerical value tells us how much a material of length L = 1 m will elongate in a given direction when heated by 1K under constant pressure.

$$\alpha^* = \frac{1}{L(T_0)}\left(\frac{\partial L(T)}{\partial T}\right)_p \tag{2}$$

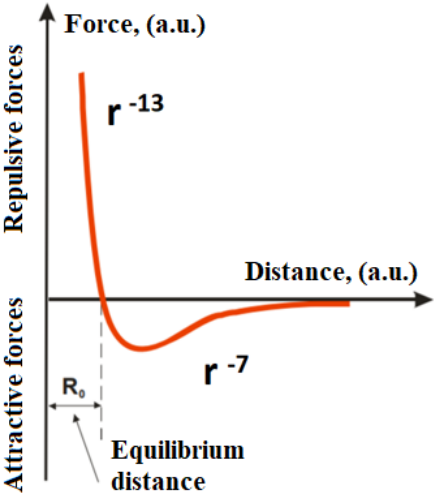


Fig. 12a. Forces acting between adjacent atoms [16]

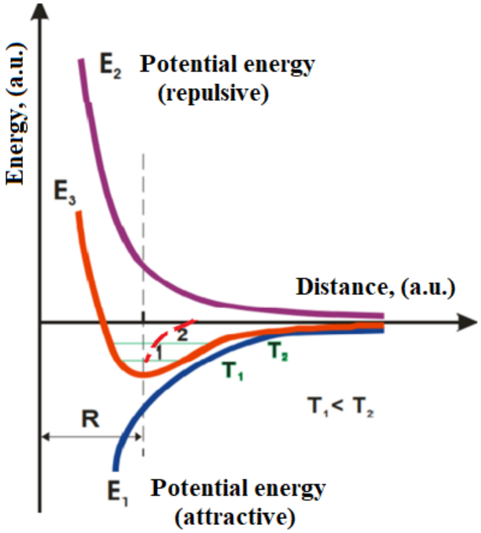


Fig. 12b. Morse curves [16]

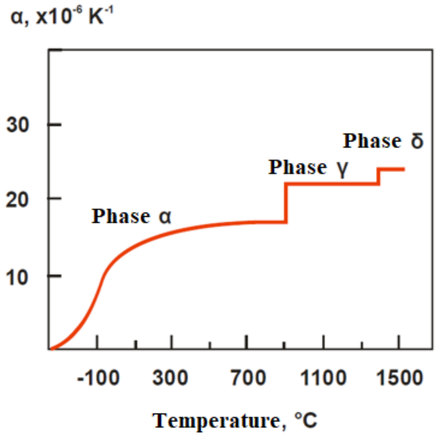


Fig. 13. Temperature dependence of CLTE for iron [16]

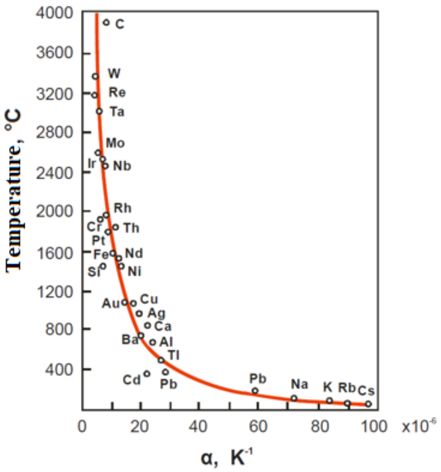


Fig. 14. Dependence of CLTE on melting point T_m (Grüneisen's law: CLTE ~ $1/T_m$) [16]

CLTE α^* in gases and liquids is, respectively, about 1,000 times and 100 times higher than CLTE in metals. The range of its numerical variation for different materials is from 10^{-2} to 10^{-7} 1/K (Fig. 15).

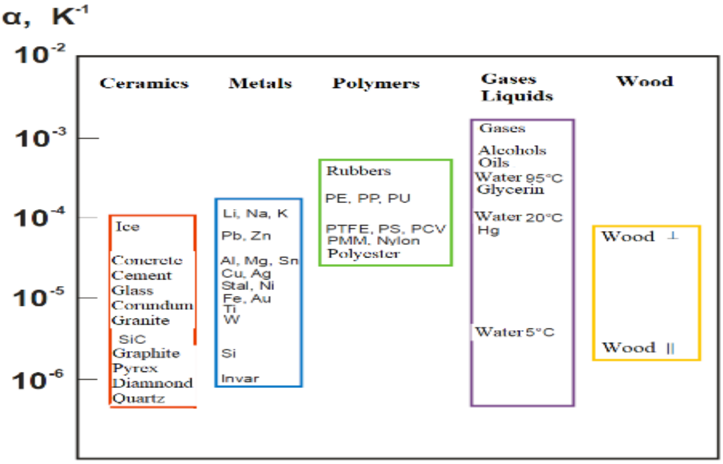


Fig. 15. Typical ranges of CLTE values of different materials [16]

4.2. Specific heat (c_V, c_p)

The capacity of a body to store heat determines its specific heat, i.e. how much heat must be input under conditions of constant volume V or constant pressure p to heat a unit mass of the body by one degree. Knowing the numerical values of specific heat contributes significantly to the determination of Helmholtz and Gibbs free energies. The values of the energies are in turn fundamental for determination of the directions of irreversible phenomena as well as the conditions of equilibrium of chemical reactions and phase transitions. Furthermore, when interpreted in terms of microscopic models and theories, they provide insights into the forces and interactions at the atomic and molecular level from which macroscopic properties of substances are then derived. Specific heat c_V is a measure of the internal specific energy u, and heat c_p is a measure of the specific enthalpy h of a body. Starting from the definition of the specific heat of a body under the conditions of the transformation $\pi = \{V, p\}$

$$c_\pi = \frac{1}{m}\left(\frac{\delta Q}{dT}\right)_\pi \tag{3}$$

and using the first law of thermodynamics for a closed system

$$\delta Q = dU + pdV = dH - Vdp \tag{4}$$

the result is

$$c_V = \left(\frac{\partial u}{\partial T}\right)_V, \quad c_p = \left(\frac{\partial h}{\partial T}\right)_p \tag{5}$$

The specific heat value is influenced by lattice vibrations and free electrons. According to the principle of energy equipartition and the Dulong-Petit law established experimentally in 1819, which states that the molar heat of chemically simple crystalline solids is independent of temperature and approximately equal to 3 universal gas constants R or approximately 25 J/(mol K), classical theory could not explain the behaviour of specific heat at low temperatures. It has been experimentally found to vary by T^3 for insulators and by T for metals [17]. This fact was only explained by Einstein and Debye on the basis of quantum mechanics. Einstein in 1907 treated lattice oscillations as a set of harmonic oscillators of equal frequency ω_{E} and quantised energy, while Debye assumed that the harmonic oscillators are not independent of each other but coupled, and the spectrum of lattice vibrations extends to a certain limiting frequency ω_{D}, which determines the characteristic Debye temperature T_{D}. Considering that when in thermodynamic equilibrium, the mean value of the energy of an atomic oscillator is subjected to Bose-Einstein quantum statistics, the relation for the molar specific heat is derived, which - for high temperature values - approaches 3R and at $T \rightarrow 0$ K, it approaches zero. The principle of energy equipartition is illustrated in Fig. 16.

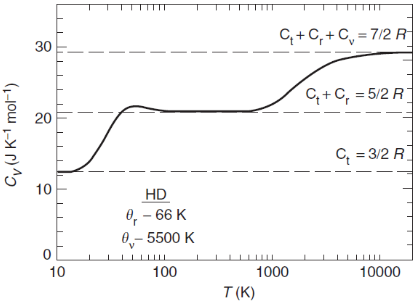


Fig. 16. Specific molar heat c_V of hydrogen deuteride H[^{2}H] [18]

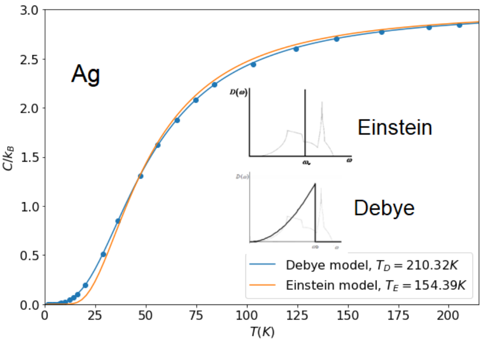


Fig. 17. Comparison of specific heat c_V measurements (points) between Debye's model (blue line) and Einstein's model (orange line) [18]

In this Figure the molar specific heat of hydrogen deuteride is a function of temperature. As the temperature increases, the contribution to the specific heat is successively translational, then rotational and finally, vibrational movements of the particle. A comparison of the specific heat c_V measurement results in silver is shown in Fig. 17.

At low temperatures, the molar specific heat c_V of silver is better approximated by Debye's model than by Einstein's. The reason is that Einstein's model does not strictly reproduce the thermal movements of the lattice. Debye's model assumes that a crystal can be treated as an isotropic elastic medium.

In this model, the wave vector and frequency are proportional to each other, and the proportionality constant is the speed of sound in that medium.

4.3. Thermal conductivity

Thermal conductivity tells us about the capacity of a given medium to conduct heat under conditions of steady-state heat transfer. In the case of insulators, phonon conductivity plays a decisive role in energy transfer. Phonon heat transfer coefficient λ_f depends on the specific heat per unit volume, c_V of the body, the mean velocity of the phonons u_f (the speed of sound), and the mean free path of phonons l_f - (6) [17]

$$\lambda_f = \frac{1}{3} c_V u_f l_f \tag{6}$$

At room temperature, $\lambda_f \sim 1/T$; at low temperatures, $\lambda_f \sim 1/T^3$. A relation similar to (6) describes the electron heat transfer coefficient. It is also worth noting the dependence of heat transfer coefficient λ_g on specific heat c_V and dynamic viscosity for gases μ and the dependence of λ_c on the mean centre-to-centre distance of the adjacent liquid molecules D and on the speed of sound propagation in the liquid u_a.

$$\lambda_g = \varepsilon\, \mu\, c_V, \quad \lambda_c = \frac{R}{N_A} \frac{u_a}{D^2} \tag{7}$$

With $N_A = 6{,}02 \cdot 10^{23}$ molecules/mol – the Avogadro number. The range of thermal conductivity coefficient includes 5 orders of magnitude (Fig. 18).

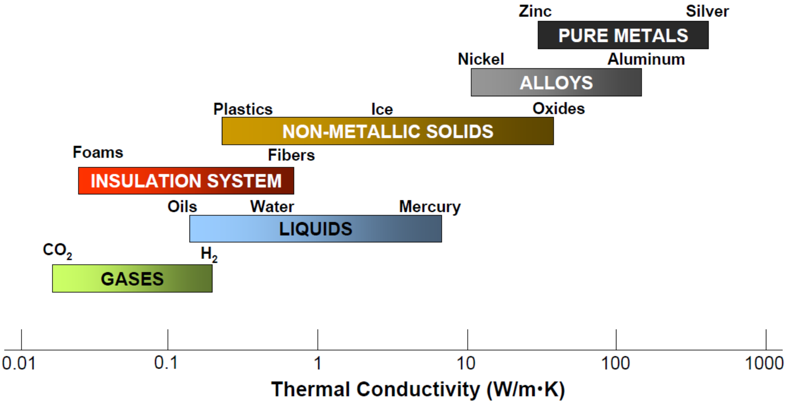
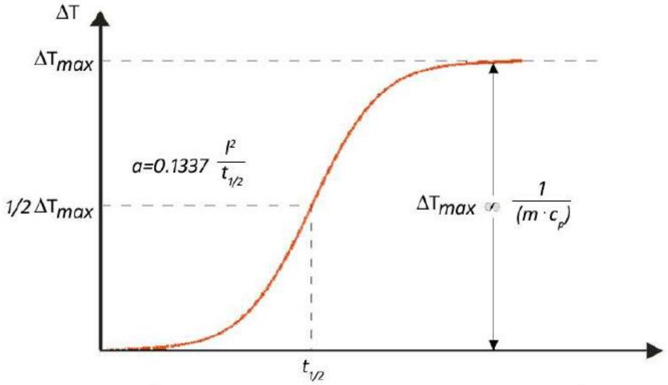


Fig. 18. Numerical ranges of thermal conductivity of gases, liquids and solids [20]

For gases (0.01 - 0.60 W/m/K) it increases with increasing temperature and is independent of pressure except for $p > 200$ MPa and $p < 2{,}666$ Pa. For liquids $\lambda = 0.08 - 0.70$ W/m/K (except for liquid metals), it decreases with increasing temperature, except for water and glycerine. For foams $\lambda = 0.02 - 0.80$ W/m/K, and for the noble metals, Ag = 420 W/m/K. Theoretically, thermal conductivity can be 8,000 W/m/K for single carbon nanotubes at room temperature [19].

4.4. Thermal diffusivity

Thermal diffusivity is also known as the temperature equalisation coefficient. This quantity can be interpreted as a material's ability to conduct heat relative to its ability to store heat. This is a very important parameter which determines the rate of equilibration of body temperature under conditions of transient heat transfer. According to the classical Parker method, to determine thermal diffusivity it is necessary to know the thickness of the sample, *l* and the half-time $t_{1/2}$, after which the excess temperature on the back surface of the sample reaches half of its maximum value (Fig. 19).



$$\lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T) .$$

Fig. 19. Concept of measuring thermal diffusivity by application of the classic Parker pulse method

5. TEST INSTRUMENTS

Thanks to the efforts of the team led by Professor Aleksander Olejnik, the Faculty of Mechatronics, Armaments and Aeronautics, together with the Warsaw University of Technology and the Institute of Aviation in Warsaw, obtained funding from the EU's European Regional Development Fund for a total amount of more than 130 million PLN, of which several dozen million PLN was assigned to the MUT Institute of Aerospace Technology.

This enabled the construction of modern laboratory facilities for joint numerical-experimental research into aircraft turbine engines. Shown in Fig. 20 is Building 66, which housed a materials storage facility; it has been refurbished and converted into a modern Aerospace Propulsion Research Laboratory. A section of this Laboratory is the Thermal Properties Testing Laboratory Unit. One of the rooms in this laboratory can be seen in the lower part of Fig. 20.

Fig. 20. View of Building 72 (DAT) and Building 66 converted into the Aerospace Propulsion Research Laboratory, with one of the rooms at the Thermal Properties Testing Laboratory

Highly dedicated testing and research equipment was purchased from the world-renowned German company NETZSCH. Figure 21 shows the Regulus STA 2500 simultaneous thermal analyser, which combines differential thermal analysis with highly accurate measurement of sample mass changes at 0.03 μg. The operating temperature range is RT ÷ to 1,000°C with a heating rate HR = 0.001 ÷100 K/min. This instrument is mainly used for determination of the initial temperature at which thermal decomposition of materials occurs.

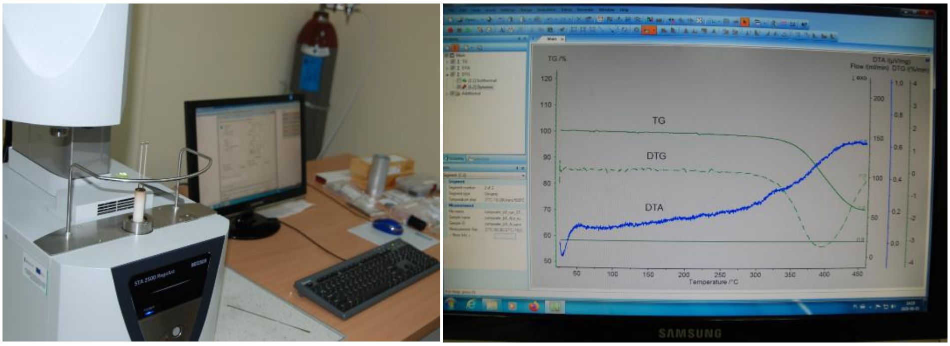


Fig. 21. View of the STA 2500 Regulus and a sample test thermogram

Figure 22 shows an LFA 467 low temperature diffusion meter with a measuring range of 0.01 to ÷1,000 mm^2/s operating in the temperature range from -100°C to +500°C. A xenon lamp was used as the source of the heat pulse. Extension of the temperature operating range from room temperature, RT, to 2,800°C is possible using the LFA 427 high temperature diffusion meter shown in Fig. 23.

Fig. 22. View of the LFA 467 low temperature diffusion meter

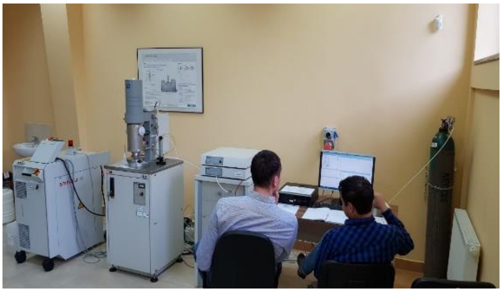


Fig. 23. View of the LFA 427 high temperature diffusion meter

Figures 24 and 25 show rod dilatometers, respectively: the low temperature DIL 402 Su Expedis with a range of -180°C to ÷ 500°C and the high temperature DIL 402 C with a range of RT to ÷ 1,600°C. In both dilatometers, the elongation of samples is measured using an inductive transducer. The change in sample length is output through a push rod to the transducer, which allows detection of sample length from 0.1 nm.

In parallel to the research into thermophysical parameters, storage dynamic modulus *E*' and loss dynamic modulus *E*" can be measured as a function of time/temperature and frequency.

The DMA 242 C dynamic mechanical analyser shown in Fig. 26 is used for this purpose. Its temperature range is -170 ÷ to 600^0 C with a heating/cooling rate of 0.01 ÷ 20 K/min, and the input frequency range 0.01 ÷ 100 Hz.

Fig. 24. View of the DIL 402 Su Expedis low temperature dilatometer

Fig. 25. View of the DIL 402 C high temperature dilatometer

The research into the thermal effects and specific heat of solids is carried out using a DSC 404 F1 Pegasus differential scanning microcalorimeter shown in Fig. 26. Specific heat is measured using the 3-curve method in this sequence: base material, sapphire and test sample. Two furnaces are used: a low-temperature steel furnace (-150^{o}C to 1,000^{o}C) and a high-temperature platinum furnace (RT to 1,500^{o}C).

Fig. 26. View of the DSC 404 F1 Pegasus microcalorimeter

Due to the complementary nature of thermophysical parameter testing and safe temperature ranges, the order of testing is important (Professor A. Panas). First, weight tests are carried out to determine the mass, m and the density of the sample, ρ at room temperature. Thermogravimetric tests are then performed. Subsequently, specific heat $c_p(T)$ and thermal effects, thermal expansion $\varepsilon(T)$, $\alpha^*(T)$, and dynamic moduli $E'(T)$, $E''(T)$, $\tan\delta\ (T)$ are tested. The tests of thermal diffusivity $a(T)$ require prior knowledge of specific heat and thermal expansion to increase the accuracy of the results.

Finally, thermal conductivity *k*(*T*) is determined as the product of density, specific heat and thermal diffusivity (Fig. 27).

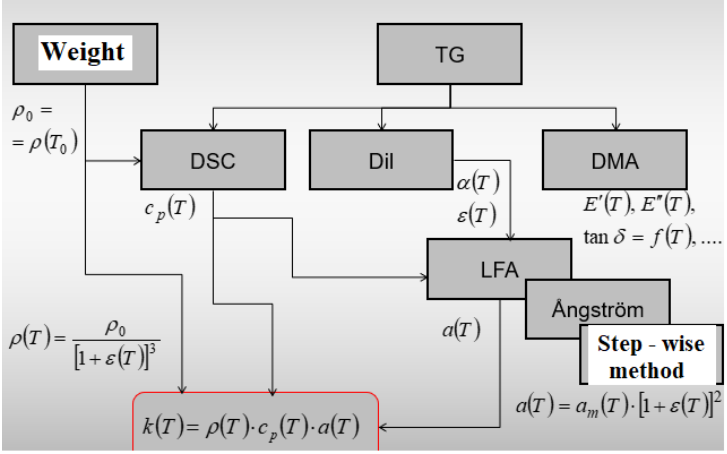


Fig. 27. Recommended sequence of testing the thermophysical parameters of solids [21]

6. SELECTED RESULTS OF PROPRIETARY RESEARCH

Developed by Professor J. Terpiłowski, the modified pulse method of thermal diffusivity of solids is based on a measurement, done with thermocouples, of the difference in temperature between the front and the back surfaces of the specimen (see the dashed curve to the left in Fig. 28), which arises as a result of absorption of a laser pulse by the specimen.

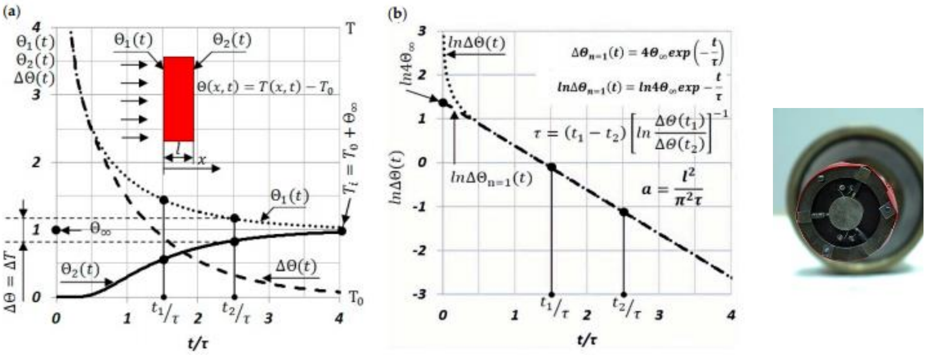


Fig. 28. Concept of the modified thermal diffusivity measurement method developed by Professor J. Terpiłowski [22]

The resulting dependence of temperature difference ΔT on time t has better metrological properties than just the temperature measured on the back surface of the sample, $T_b(t)$, and with an appropriate choice of the time window width, in which the model temperature difference is fitted by means of a nonlinear regression method, it allows determination of the thermal diffusivity with an uncertainty of a few per cent.

The results of thermal diffusivity research on Fe-Ni alloys obtained recently have been published in prestigious scientific journals - the MDPI *Materials* and the *Journal of Alloys and Compounds.* (Figs. 29a, 29b)

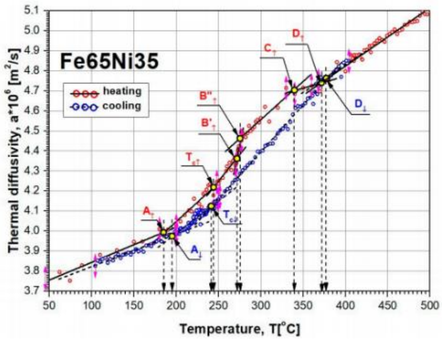


Fig. 29a. Results of thermal diffusivity tests of Fe65Ni35 alloy with the modified pulse method [22]

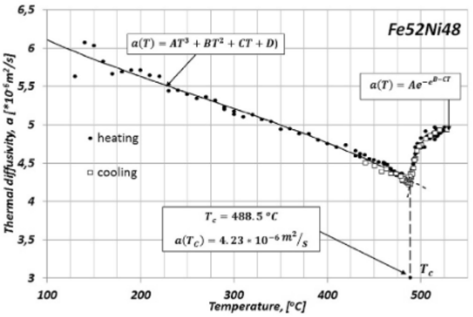
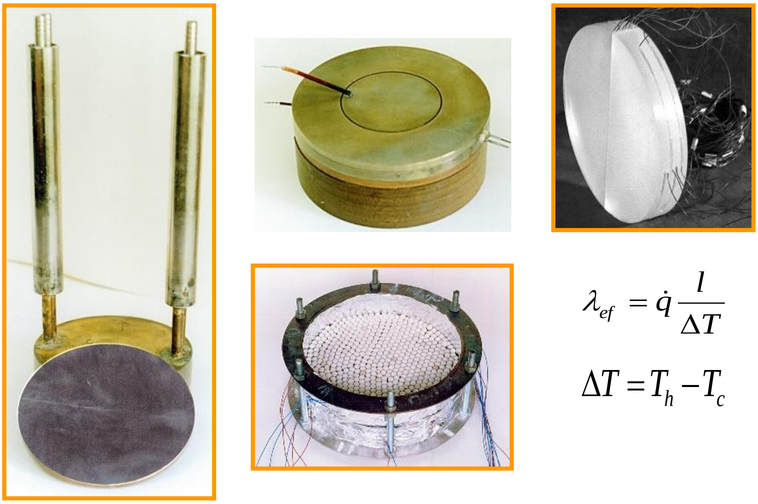


Fig. 29b. Results of thermal diffusivity tests of Fe52Ni48 alloy with the modified pulse method [23]

The test stand for thermal conductivity of insulating materials in the temperature range of RT to 700^{0}C, developed and built by Professor P. Koniorczyk, enables testing the effects of reducing thermal conductivity in construction materials. Fig. 30 shows a cooler (radiator) with a bifilar coolant flow, the electric primary and protective heaters, a cylindrical specimen of diameter $\phi = 200$ mm with thermocouples attached, and a fixture for the specimens, which are in the form of ceramic spheres. The measurement of the effective thermal conductivity λ_{ef} requires knowledge of the sample thickness l, the temperature difference ΔT between a hot T_h and a cold T_c copper plate adhering to the surface of the specimen, and the heat flux density q.

Example results of tests of effective thermal conductivity of white and dotted white expanded polystyrene from Austrotherm as a function of sample thickness l are shown in Fig. 31. The effect of reduced thermal conductivity associated with the coupling of different heat transfer mechanisms (conduction plus radiation) is evident here. The results of this research were obtained in the course of the doctoral dissertation of Anna Kondrot-Buchta, M.Sc., defended in 2014 and supervised Professor P. Koniorczyk.



$$\lambda_{ef} = \dot{q}\,\frac{l}{\Delta T}$$

$$\Delta T = T_h - T_c$$

Fig. 30. Selected components of the test stand for thermal conductivity of insulating materials, developed by Professor P. Koniorczyk [6, 7]

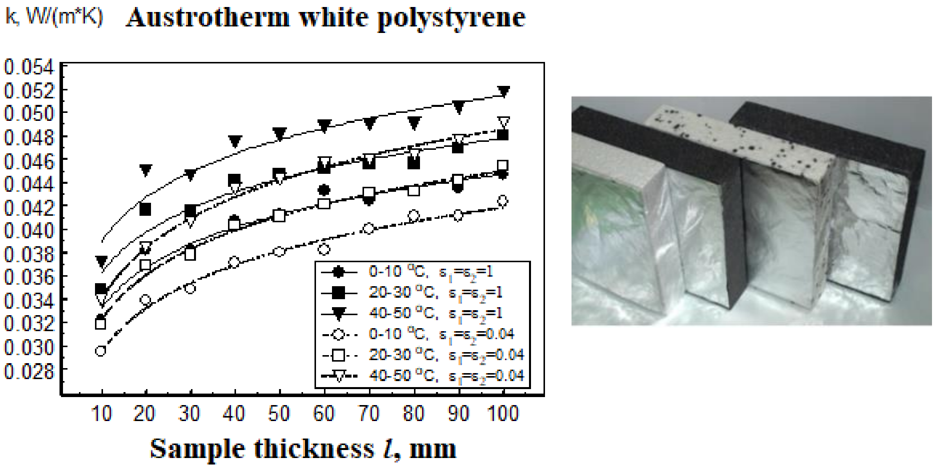


Fig. 31. Experimental results of thermal conductivity *k* of white EPS and white dotted EPS as a function of sample thickness [24]

A unique approach to the simultaneous identification of specific heat, thermal conductivity and - with a known density - thermal diffusivity, is the continuous heating method illustrated in Fig. 32.

A description of the method and its experimental validation on the example of duralumin alloy PA6 have been published in the prestigious journal, *Thermochimica Acta* in 2019.

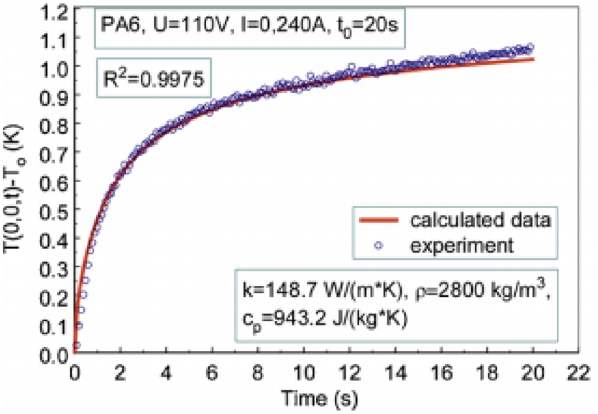


Fig. 32. Results of simultaneous identification of specific heat and thermal conductivity of the PA6 alloy by continuous heating [12, 13]

The results of the verification of specific heat and thermal conductivity of PMMA and PA6 insulating materials obtained with the inverse coefficient method and with the DSC 404 F1 Pyris 1 microcalorimeter (for determination of specific heat) as well as with the Poensgen plate apparatus (for determination of thermal conductivity) are shown in Fig. 33.

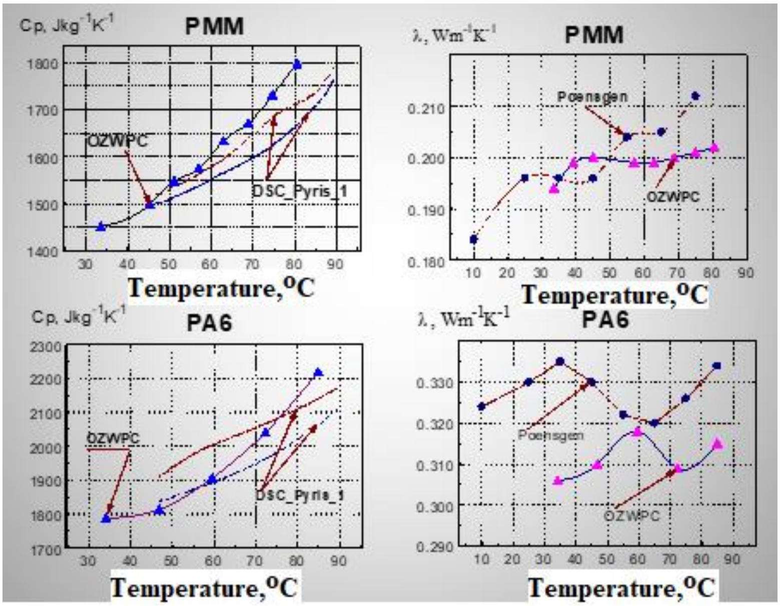
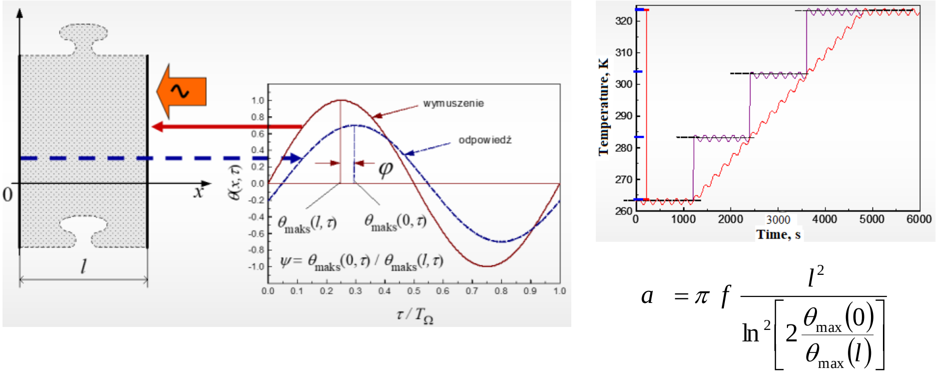


Fig. 33. Experimental verification of the results of the identification of thermophysical parameters of PMMA and PA6 with the inverse coefficient method [14]

An interesting solution proposed by Professor A. Panas in 2005 is an experimental test stand for determination of thermal diffusivity with the modified Ångström method (Fig. 9, 34).



$$a = \pi f \frac{l^2}{\ln^2\left[2\frac{\theta_{\max}(0)}{\theta_{\max}(l)}\right]}$$

Fig. 34. Concept of measuring thermal diffusivity by thermal periodic input [25]

The concept of measuring thermal diffusivity *a* is to measure the amplitude $\theta_{\max}(l,\tau)$ and the phase shift φ at a given point distant by *l* from the surface to which thermal inputs are supplied with a period of $T_\Omega = 2\pi/f$. Peltier modules were used for this purpose. The results of measuring the thermal diffusivity of water, supercooled water and ice are shown in Fig. 35. Achieving a thermal resolution of thermal diffusivity this high was possible by superposition of a linear temperature rise with periodic input function.

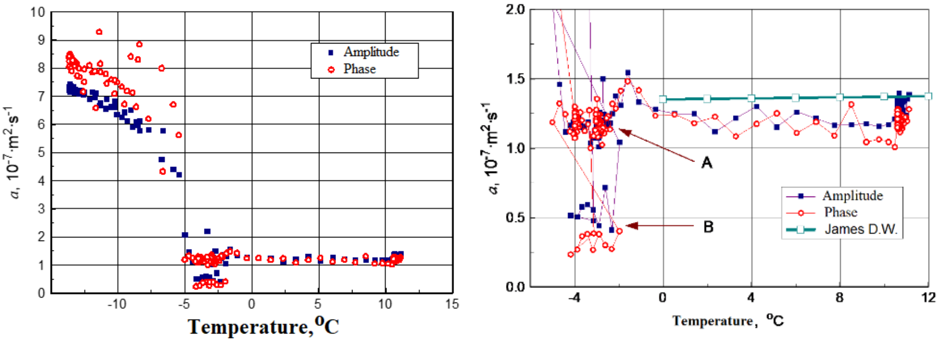


Fig. 35. Results of thermal diffusivity tests on water and ice using the modified Ångström method [26]

A unique method for testing thermal diffusivity of solids under conditions of systematic heat exchange, based on the determination of characteristic times of a specimen immersed successively in two fluids of similar thermal properties (e.g. water and alcohol) and temperatures (e.g. 5 and 10°C) was proposed by Professor A. Panas in 2002.

The test stand and the results of PMMA thermal diffusivity tests using this method are shown in Fig. 36.

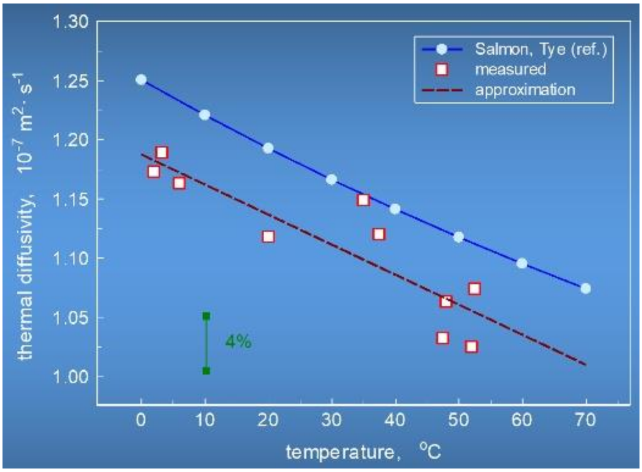


Fig. 36. Results of the research into thermal diffusivity of PMMA by the step function method under conditions of systematic heat exchange (A. Panas 2002)

Another example of the proprietary design solutions is the test stand developed by Professor A. Panas in 1985 (Fig. 7) with a specially designed interferometer (Fig. 37). It is used to examine the thermal expansion of solids using an interference method with a high thermal resolution ranging from -120^{0}C to approximately 1,300^{0}C. By directly measuring the temperature of the specimen and using virtual instrument technology, it is possible to perform extremely accurate tests on phase transition and direct measurement of specimen temperature.

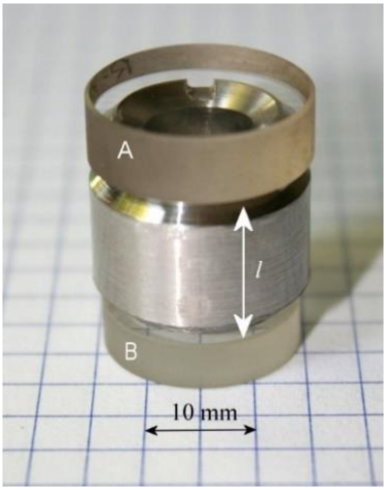


Fig. 37. Overview of the interferometer (A. Panas, 1985)

Example results of coefficient of linear thermal expansion (CLTE) measurements of ARMCO iron and FeNi40 alloy are shown in Figs. 38 and 39.

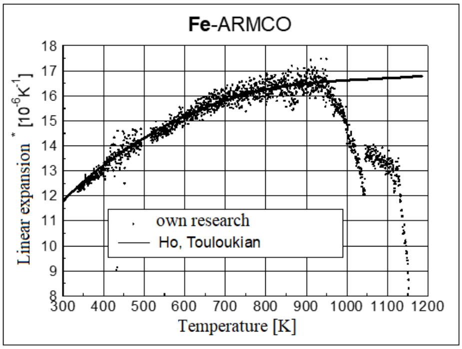


Fig. 38. Fe-ARMCO CLTE test results (A. Panas)

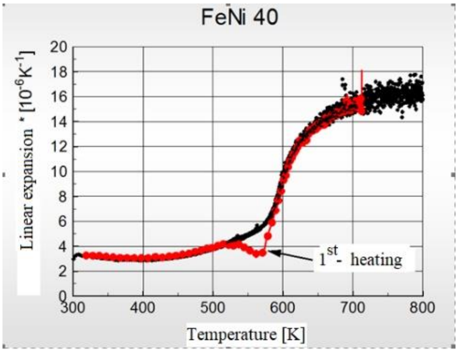


Fig. 39. CLTE test results for Fe60Ni40 alloy in the first and second heating runs (A. Panas)

The results of thermal effects, specific heat, thermal decomposition and temperature and heat flux density measurements of a Bolsius paraffin wax for the application of the material in construction of a passive cooling system for high-power LED panels were published in the *International Journal of Thermophysics* in 2017 (Fig. 40).

Figures 41 and 42 show the results of thermal diffusivity, specific heat and thermal expansion tests on several gun steel grades using LFA 427 and LFA 467 diffusion meters. The use of a DSC 404 F1 microcalorimeter and a DIL 402 C dilatometer was forced by the determination of the temperature range of the ferrite-austenite phase change, contributing to the cracks formed in machine gun barrels after a series of shots. The results of the experimentally obtained research were published in the prestigious journal MDPI *Metals* in 2020.

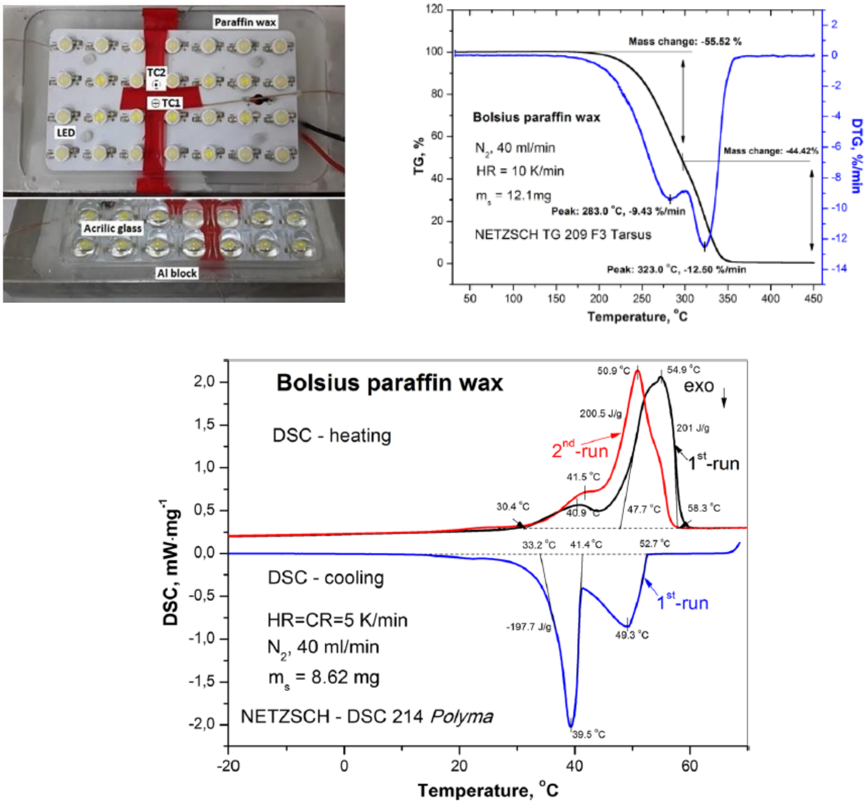


Fig. 40. Thermogravimetric results and thermal effects of Bolsius paraffin wax for passive cooling of high power LED panels [27]

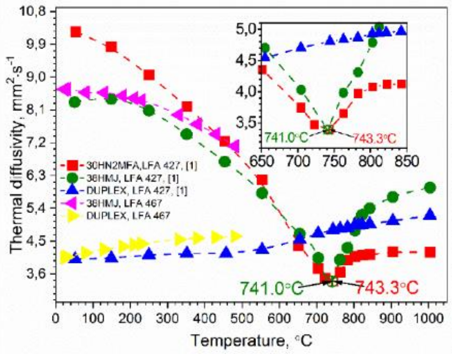


Fig. 41. Test results of thermal diffusivity of gun steel grades 30HN2MFA, 38HMJ and DUPLEX with the LFA427 and LFA467 diffusion meters [28]

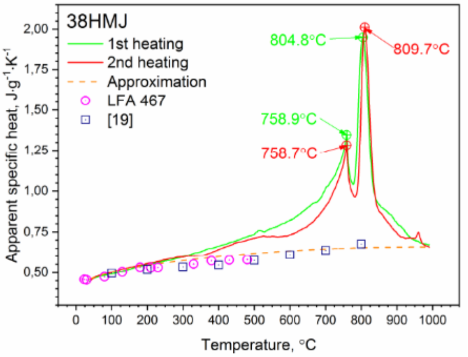


Fig. 42. Test results of the apparent specific heat of 38HMJ gun steel with the DSC 404 F1 and the LFA467 [28]

Interesting results from DMA mechanical modulus studies of double-base solid rocket propellants were used to determine the temperature range of glass transition and activation energy. Figure 43 shows example results of the *E*’, *E*” and tan δ modules for PAC fuel, derived from Marcin Cegła’s PhD dissertation defended in 2017 with this author as the supervisor.

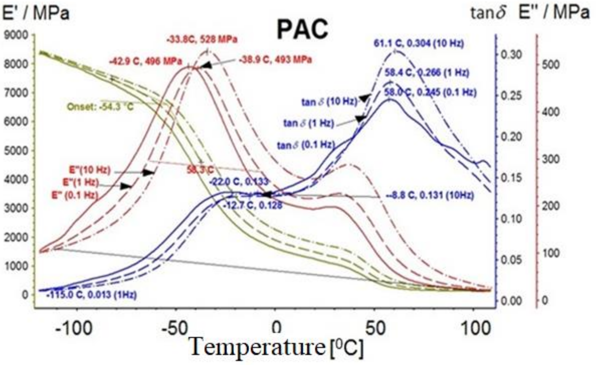


Fig. 43. DMA test results for PAC solid rocket propellant [29]

7. CONCLUSIONS

This paper presented selected achievements of the Thermodynamics Team, which has been a part of the Department of Aerodynamics and Thermodynamics at the Faculty of Mechatronics, Armament and Aeronautics of the Military University of Technology (MUT) for the last 40 years in the area of thermomechanical properties of solids and liquids. With a wide audience in mind, especially the first-year students starting the New Academic Year 2020/2021 at the Department, the genesis of the team’s undertaking of research into thermal properties, with temperature measurements and numerical simulations of temperature and stress fields, was presented. Basic heat transfer modalities and thermophysical parameters are discussed next. Further on, test stands are characterised – both those commercially purchased in the recent past and those developed in-house, along with modifications made to the existing test methods. In the final section, selected results of studies of thermophysical parameters of solids and liquids and DMA studies of dynamic properties of solid rocket propellants are shown. The information provided here may prove useful to MSc students, PhD students and all those considering research into thermal properties of solids.

ACKNOWLEDGEMENT

The author wishes to thank his fellow Professors: Janusz Terpiłowski, Piotr Koniorczyk, Andrzej Panas and Marek Preiskorn, PhD for disclosing the results of their work and for consenting to their publication.

FUNDING

The author received no financial support for the research, authorship, and/or publication of this article.

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Właściwości cieplne ciał stałych – podstawy teoretyczne, metody badań, wybrane wyniki badań własnych

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Streszczenie. Artykuł nawiązuje do wykładu inauguracyjnego opracowanego przez autora w związku z inauguracją Nowego Roku Akademickiego 2020/2021 na Wydziale Mechatroniki, Uzbrojenia i Lotnictwa WAT w dniu 2 października 2020 r. Przedstawiono genezę badań właściwości cieplnych ciał stałych prowadzonych od połowy lat siedemdziesiątych XX w. przez pracowników części termodynamicznej Zakładu Aerodynamiki i Termodynamiki, a w dalszej kolejności podstawowe mody wymiany ciepła, podstawy teoretyczne rozszerzalności cieplnej, ciepła właściwego, przewodności cieplnej i dyfuzyjności cieplnej ciał stałych. Pokazano aparaturę pomiarową powstałą w wyniku realizacji własnych opracowań naukowych oraz zakupioną od wiodących na rynku producentów jak również przedstawiono wybrane wyniki badań właściwości cieplnych ciał stałych, które w dużej mierze są efektem zastosowania własnych procedur badawczych.

Słowa kluczowe: wymiana ciepła, parametry termofizyczne, procedury badawcze, stanowiska pomiarowe