# Thermal Capacity Measurement of Engineering Alloys in Dependence on Temperature.

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**ABSTRACT**

Thermophysical laboratory at Department of Thermal Engineering of Technical University of Ostrava deals with basic thermophysical property measurements of engineering alloys. The properties measured are thermal conductivity, electrical resistivity, thermal capacity, thermal expansion, phase temperature transformation, Curie temperature, density, thermal diffusivity, electrical conductivity and radio frequency penetration depth.

This paper deals with measurement of thermal capacity of electrically conductive materials. Combined furnace-direct resistance heating of sample is employed. Radiance heat transfer is reduced by proper shape of sample. Heat convection is eliminated by means of vacuum. The system is put into vacuum chamber. Vacuum is generated by two-stage rotary oil pump (back vacuum) and turbo molecular vacuum pump (high vacuum). Back vacuum is measured by Pirani vacuometer, high vacuum is measured by Penning vacuometer. Vacuum also prevents the specimen from oxidation at high temperature. Sample is put into special furnace and its temperature is programmable. Temperature of the sample is measured by welded thermocouple. An electric rectangular pulse is brought into the sample and electric energy is changed directly into heat in sample volume. Amount of heat is calculated from pulse width, current and voltage across sample section. Voltage is sensed by probes welded in distance *s* on the sample. Current is measured by standard shunt. Specific thermal capacity (specific heat) is calculated from supplied heat, temperature rise and mass of the sample. Electrical resisitivity and thermal conductivity can be calculated as well.

**KEY WORDS:**

electrical resistivity; engineering alloys; specific heat; thermal conductivity; thermophysical properties.

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## 1. INTRODUCTION

Different materials are used for making of engines, instruments, tools and equipment in mechanical and electrical engineering, metallurgical and chemical industry. Basic most important materials are still metals and alloys. For structures, which are considerably loaded at high temperatures there are used at present the materials which are not based on iron but nickel or cobalt, in addition to heat resistant steels.

All thermophysical properties come from basic parameters of crystal lattice and structure; theoretical fundamentals of the properties are elaborated into different profundity. Designer selects proper alloy, required dimensions and processing method by one basic property or whole properties. He or she can come out from experience or theoretical calculation but he or she has to known principal of basic properties and connect it with concrete details for different metals and alloys. Only in this way designer can choose the right alloy, put to use optimally its property and properly determine processing method.

*Specific heat* determines heat, which is led into (taken away from) mass unity of given substance to heat up (cool down) by one degree (K). This thermophysical parameter is indicator of metals and alloys heating easiness. Therefore is inevitable to know specific heat of required material for thermal-technical calculations. Metals with the highest specific heat at room temperature are beryllium, magnesium alloys and aluminium; then follow iron and other metals. Platinum, gold and lead have least specific heat.

Specific heat of magnetic materials is changing significantly by coupling among elementary magnets. Specific heat under magnetization is more increasing with temperature compared to the same material without effect of external magnetic field. Positive magnetic anomaly of specific heat is expression of energy needed for disarrangement of exchange coupling among elementary magnets. The influence of magnetization suddenly expires at Curie temperature. Specific heat is different at heating under constant magnetization and constant magnetic field; specific heat under constant magnetic field is greater [1].

Knowledge of *electrical resistivity* and *permeability* is necessary for calculation of electromagnetic energy penetration depth at induction heating. Only electrically conductive materials can be heated by induction heating. Theory of induction heating is based on electrodynamics resulting from Maxwell´s equations. Induction heating makes possible extra high specific absorbed power into metal charge. On the base of knowledge of permeability and electrical resistivity, temperature dependence and supply frequency selection is possible to determine distribution of heat generated in metal charge. Induction heating therefore gains even wider range of exercise in metallurgy and mechanical engineering [2].

Specific heat can be obtained directly from the measured quantities by adiabatic, differential scanning, modulation or pulse calorimetric techniques.

Adiabatic calorimetry is versatile technique for the direct measurement of specific heat from low to moderately high temperature. This method is the most precise and sensitive. Heat loss is eliminated by adiabatic shield so there is no need to introduce any corrections in moderate temperature range. The adiabatic conditions are not easy to obtain at high temperature. Electronic control system is complicated.

Differential scanning calorimetry is perfect for small samples. It can be used for milligram quantities over a temperature range of 100 to 1000 K. The method is simple and rapid, but reference material is needed and interpretation of complex curves is not easy.

Modulation calorimetry employs periodically modulated power which heats the sample and temperature changes from mean value are registered. The temperature changes can be small, so heat loss can be neglected. The method can be used in wide temperature range.

Pulse calorimetry can be classify into different categories by initial temperature conditions of sample and its final temperature. The initial temperature can be achieved by furnace or by direct resistance heating of the sample. The temperature change of sample from steady state condition in the first technique is about 1 K so the radiation losses need not be taken into account. The method can be used only for electrically conductive materials [3, 4].

## 2. METHOD

At Department of Thermal Engineering specific heat capacity is measured by pulse method. Strip sample bended to meander shape is put into temperature programmable furnace. Block diagram of the experimental apparatus is shown in Fig. 1. Detail of temperature and voltage sensing is in Fig. 2. Accurately defined electric pulse is brought into the sample. Battery is used as power-supply. Timer and power switch defines the pulse width. Sample temperature is sensed by thermocouple up to 1 600 °C. *Specific heat capacity* is calculated from temperature difference, pulse energy and sample mass. Current is measured by standard shunt, voltage across the sample is sensed by voltage probes. *Electrical resistivity* is calculated from the acquired data as well.

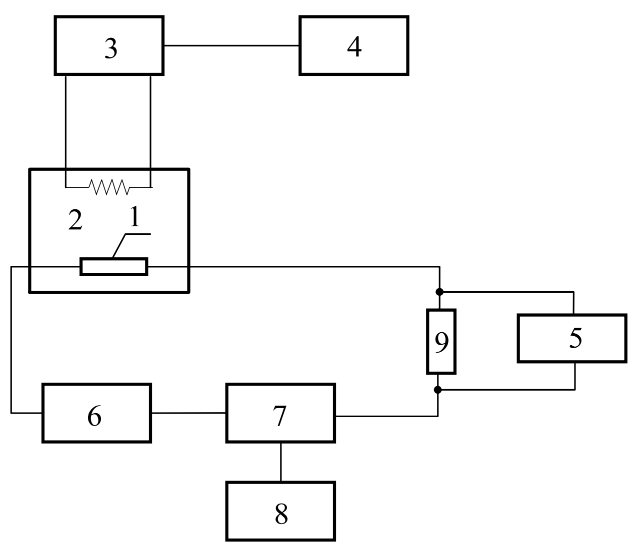


**Fig. 1.** Block diagram of experimental apparatus. 1 – sample, 2 – furnace, 3 – programmable power supply, 4 – controller, 5 – data logger, 6 – battery, 7 – power switch, 8 – timer, 9 – standard shunt.

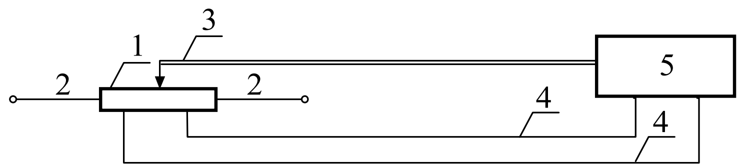


**Fig. 2.** Detail of temperature and voltage sensing. 1 – sample, 2 – electric energy supply leads, 3 – thermocouple, 4 – voltage probes, 5 – data logger.

Sample is put into vacuum chamber for elimination of heat losses by convection and is bended like meander to reduce radiation losses. A thermocouple is welded in sample center and voltage probes are welded symmetrically on both sides, Fig. 3.

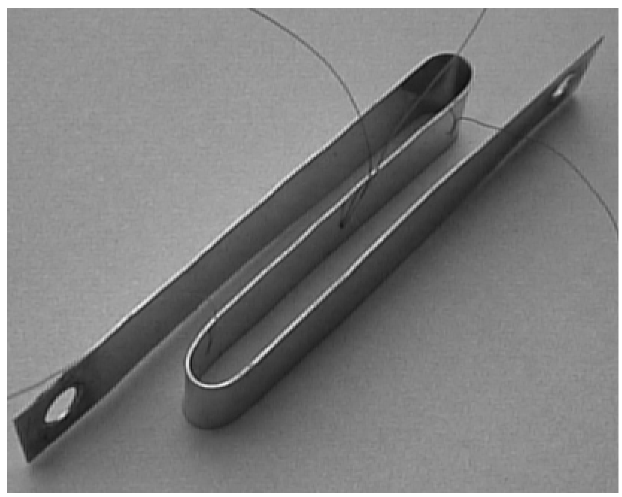

**Fig. 3.** Sample with thermocouple and voltage probes.

Specific heat is calculated from equation

$$c_p = P \cdot \tau / ( m \cdot \Delta t) \tag{1}$$

where $c_p$ is specific heat ( $J \cdot kg^{-1} \cdot K^{-1}$ ), $P$ – power (W), $\tau$ - time (sec), $m$ – mass of the section (kg), $\Delta t$ - temperature difference (K).

Temperature dependence of electrical resistivity can be calculated from stored data as well, because basic principal of the measurement is the same as four-probe contact method. It can be calculated from equation

$$\rho_e = ( A / s) \cdot ( V / I ) \tag{2}$$

where $\rho_e$ is electrical resistivity ($\Omega \cdot m$), $A$ – sample cross section area ($m^2$), $s$ – distance of voltage probes (m), $I$ – electric current (A), $V$ – voltage across distance $s$ (V).

Electrical conductivity is the reciprocal of resistivity. It is given by the ratio of current density, $J$, to electric field strength, $E$,

$$\kappa = j / E \tag{3}$$

where $\kappa$ is electrical conductivity ($S \cdot m^{-1}$), $j$ – current density ($A \cdot m^{-2}$), $E$ – electric field strength ($V \cdot m^{-1}$).

There is possible to calculate thermal conductivity by Wiedemann – Franz relation from electrical resistivity by eqution

$$\lambda = L \cdot (T / \rho_e) + C \tag{4}$$

where $T$ is thermodynamic temperature (K), $\rho_e$ – electrical resistivity ($\Omega \cdot m$), $L$ – modified Lorenz number ($V^2 \cdot K^{-2}$), $C$ - constant which represents all contributions other than those due to electrons ($W \cdot m^{-1} \cdot K^{-1}$), Reference [5].

## 3. EXPERIMENTAL MEASUREMENT

Measurement was done on sample from stainless steel with chemical composition (% by weight): C - 0.07; Cr - 18; Ni - 9.5; Mn - 0.9 in temperature span 20 – 800 °C.

*Calculation example* of specific heat, electrical resistivity a thermal conductivity at 20 °C.

These data were received by experimental measurement:

- sectional voltage $V_{sekc} = 0.175$ V,
- ample heating current $I = 33.364$ A,
- temperature rise $\Delta t = 4.493$ K,
- pulse width $\Delta\tau = 0.421$ s.

*Specific heat* was calculated by equation (1), where power was calculated by formula

$P = V_{sec} \cdot I$

$P = 0.175 \cdot 33.364$

$P = 5.838$ W

Mass of the sample was weight before measurement

$m = 0{,}00128$ kg

from that specific heat

$c_P = P \cdot \tau / (m \cdot \Delta t)$

$c_P = 5.838 \cdot 0.421 / (0{,}00128 \cdot 4.493)$

$c_P = 427.095\ J \cdot kg^{-1} \cdot K^{-1}$

*Electrical resistivity* was calculated from equation (2), where sectional area

$A = v \cdot h$

where $h$ is sample thickness, $v$ – sample height (width of the strip)

$A = 0.0004 \cdot 0.0101$

$A = 4.040.10^{-6}\ m^2$

s – distance of probes was 0.040 m; thermal expansion is not taken into account, from that electrical resistivity

$\rho_e = ( A / s) \cdot ( V / I )$

$\rho_e = (4.040 \cdot 10^{-6} / 0.040) \cdot (0.175 / 33.364)$

$\rho_e = 5.301 \cdot 10^{-7}\ \Omega \cdot m$

*Thermal conductivity* was calculated from equation (4)

$\lambda = L \cdot (T / \rho_e) + C$

where $L$ is modified Lorenz number and $C$ - constant which represents all contributions other than those due to electrons, Reference [5],

$\lambda = 2.39 \cdot 10^{-8} \cdot (293.15 / 5.301 \cdot 10^{-7}) + 4.2$

$\lambda = 17.\ 417\ W \cdot m^{-1} \cdot K^{-1}$

Results of measurement of specific heat, electrical resistivity and thermal conductivity (steel DIN 1.4301) are in Table I.

**Table I.** Results of measurement.

| **temperature (°C)** | **specific heat ($J.kg^{-1}.K^{-1}$)** | **resistivity·$10^7$ ($\Omega \cdot m$)** | **thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)** |
|---|---|---|---|
| **20** | 427.095 | 5.301 | 17.417 |
| **30** | 426.692 | 5.321 | 17.816 |
| **100** | 457.712 | 5.925 | 19.252 |
| **200** | 481.247 | 6.644 | 21.221 |
| **300** | 503.790 | 7.262 | 23.062 |
| **400** | 554.469 | 8.175 | 23.879 |
| **500** | 594.478 | 8.725 | 25.378 |
| **600** | 641.646 | 9.151 | 27.005 |
| **700** | 669.269 | 9.446 | 28.823 |
| **800** | 691.523 | 9.701 | 30.638 |

Measured values were compared with literature data of steel with similar chemical composition (% by weight) C - 0.15; Cr - 18; Ni - 9; Mn - 0.5; Si - 0.8, Reference [6]. Dependence of specific heat on temperature for measured steel (curve 1) and literature data (curve 2) is shown in Fig. 4. Dependence of electrical resistivity on temperature for those steels is shown in Fig. 5, dependence of thermal conductivity on temperature for those steels is shown in Fig. 6, measured steel (curve 1) and literature data (curve 2), Reference [6].

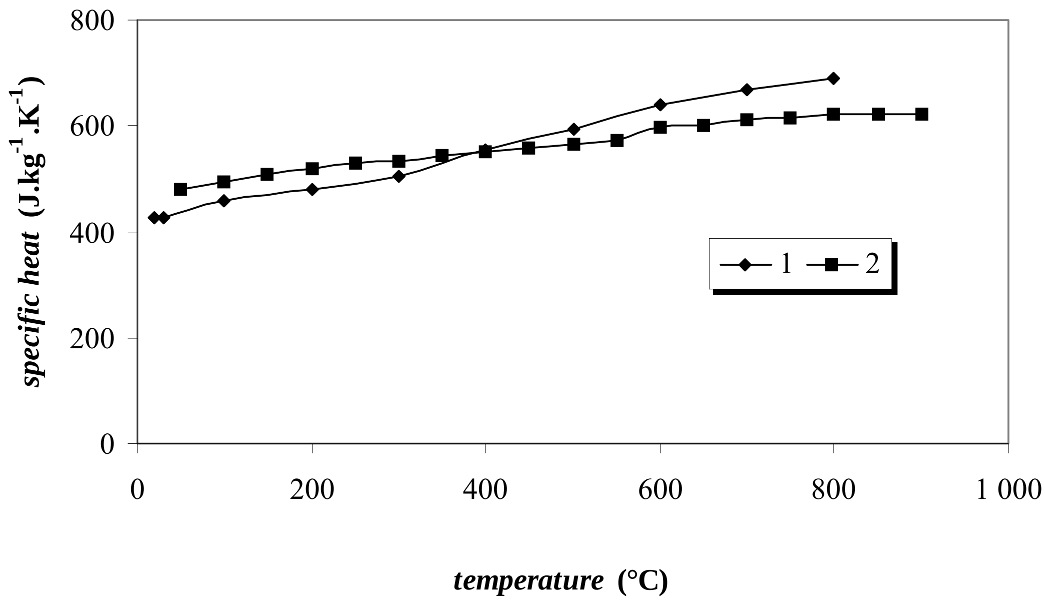


**Fig. 4.** Dependence of specific heat on temperature.

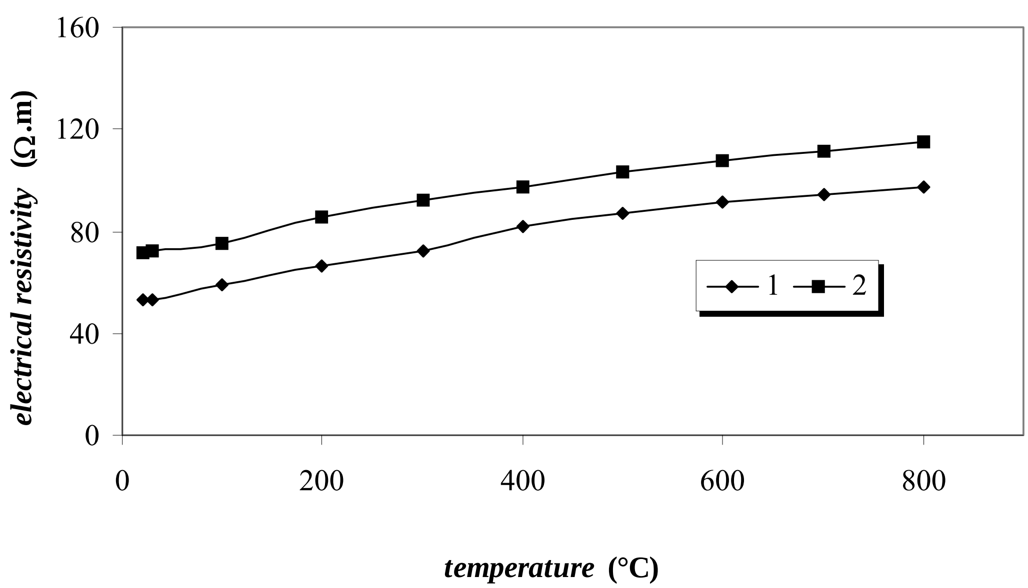


**Fig. 5.** Dependence of electrical resistivity on temperature.

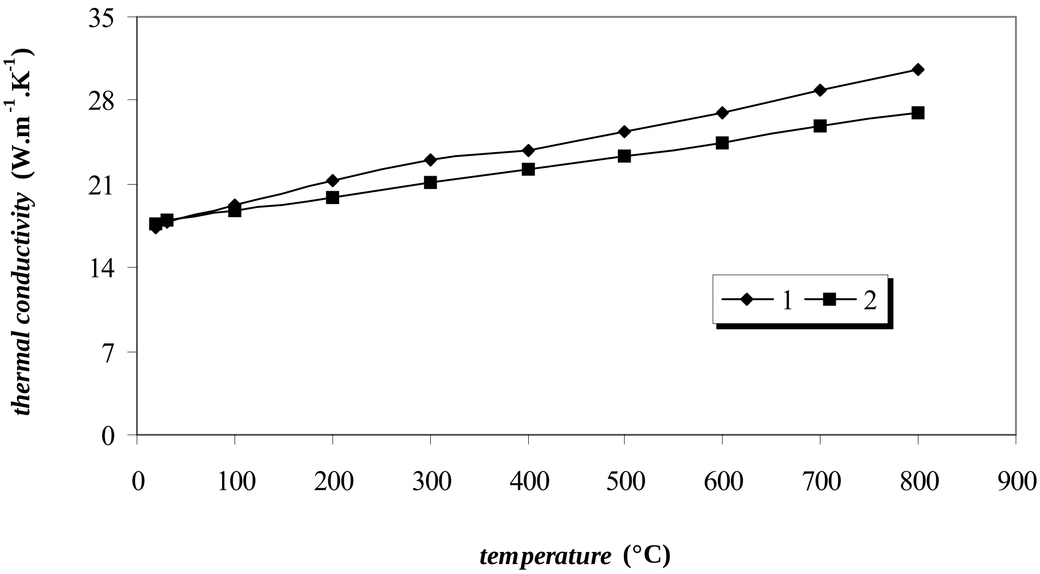


**Fig. 6.** Dependence of thermal conductivity on temperature.

## 4. CONCLUSION

Specific heat capacity was measured in our laboratory by multiple property apparatus. Sample in shape of rod was direct resistance heated. Adiabatic condition were achieved by means of vacuum and cylindrical mirror heated in the same way like sample. Control system was complicated because $\varepsilon$ (emissivity coefficient) of the mirror was different than $\varepsilon$ of sample. So we developed simpler method of specific heat capacity measurement.

The new method employing meander shape of sample is simpler and even if condition are not completely adiabatic results of measurements are good. Temperature rise of sample is only a few Kelvin, width of heating pulse is short and substantial part sample radiation is compensated by the meander shape so heat losses need not be taken into account.

## ACKNOWLEDGMENT

This research was supported by Grant Agency of the Czech Republic, Grant ID No. 106/03/D200 and 106/03/0264.

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