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Comparative experimental studies of the specific heat of Fe and Ni using scanning calorimetry methods

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Abstract

This study presents comparative experimental investigations of the specific heat of materials undergoing phase transitions, with the aim of assessing the capabilities and metrological limitations of differential scanning calorimetry methods and instrumentation. Nickel and iron were selected as reference materials due to the availability of reliable literature data for verification. Two approaches to determining heat flux density in differential scanning calorimetry were compared using three calorimeters: Pyris 1 (PerkinElmer), DSC 3 (Mettler Toledo), and DSC 404 F1 Pegasus (Netzsch). Measurements were conducted in a protective atmosphere over a temperature range up to 600°C on samples that had been thermally stabilised prior to testing. For the Pyris 1 calorimeter, a proprietary measurement procedure based on heating and cooling cycles with isothermal holds was applied, enabling reliable determination of specific heat during heating and cooling. In contrast, for the DSC 3 and DSC 404 F1 instruments, the analysis was primarily based on heating data. The obtained results allowed analysis of the second-order phase transition in nickel, including determination of the transition end temperature and the temperature corresponding to the maximum differential scanning calorimetry peak. The phase transition in iron was characterised solely using data from the DSC 404 F1 instrument, due to measurement limitations of the remaining equipment. Additionally, three independent temperature-dependent specific heat characteristics were compared, obtained using a relative method with identical reference standards and standard differential scanning calorimetry signal conversion procedures. The results contribute to improved understanding of calorimetry methodology and measurement uncertainty in materials undergoing phase transitions.

Keywords: Specific heat capacity; Ferromagnetic phase transition; Heat-flow calorimetry; Power-compensation calorimetry

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1. Introduction

The specific heat of materials is one of the basic thermo-physical properties, important both from the point of view of scientific research and engineering applications [1,2]. Accurate knowledge of this quantity allows for effective modelling of thermal processes, optimisation of technological parameters, and design of materials and devices operating in variable temperature conditions. In the case of metals undergoing phase transitions, such as iron (Fe) and nickel (Ni), it is particularly important to take into account changes in specific heat as a function of temperature and the characteristics of these transitions, and research has been conducted for almost 100 years [3,4].

Differential scanning calorimetry (DSC) is one of the most commonly used methods for testing specific heat [5−9]. It is characterised by high sensitivity, the ability to perform measurements over a wide temperature range, and relatively simple laboratory procedures with an error of less than 3% [8,10]. Despite the popularity of this method, the results obtained may vary depending on the apparatus used, the measurement methodology

Nomenclature

c_p – specific heat capacity, J/(kg·K)
f – thermogram data
m – mass, kg
t – time, s
T – temperature, °C

Subscripts and Superscripts
base – baseline date
C – Curie
ref – reference
sp – specimen

Abbreviations and Acronyms
DSC − differential scanning calorimetry

and the method of data processing, in this case, thermograms. It is therefore advisable to conduct comparative studies to assess the consistency and precision of specific heat measurements performed with different devices and techniques within the DSC method.

This article presents the results of comparative experimental studies of the specific heat of iron and nickel, conducted using three different scanning calorimeters: PerkinElmer Pyris 1, Mettler Toledo DSC 3 and Netzsch DSC 404 F1 Pegasus. Iron and nickel were selected due to their ferromagnetic properties at room temperature and their loss in the second type of phase transition after exceeding the characteristic Curie temperature. Iron is additionally characterised by the occurrence of a first-order transition. These phenomena make it possible to use the characteristic temperature as an additional metrological parameter. The analysis examined the impact of measurement conditions and declared temperature change programmes, in particular the use of isothermal stops, on the measurement results. In addition to mutual comparison, the consistency of the obtained results with the literature data was also checked. Particular attention was paid to the characteristics of second-order phase transitions and the assessment of the metrological capabilities and limitations of individual devices.

Regardless of the specific objectives mentioned above, the research project is based on general methodological principles that focus on key issues across the entire field of modern thermal analysis. Very often, the measurement accuracy reported by authors is simply a repetition of data provided by manufacturers. The problem is that this information mainly concerns the repeatability of measurements and compliance with reference data obtained under specific experimental conditions. Not all of these conditions can be maintained during routine experiments. The results of the round-robin tests described in this article are intended to indicate the reliability of measurement results obtained under typical conditions, in terms of both qualitative and quantitative data.

2. Research equipment and subject of study

Although one might initially think that the research issue described above is limited solely to the problem of equipment, it should be noted at the outset that the measuring equipment was selected in such a way as to represent three different types of methods for implementing the idea of scanning calorimetry [11]. The Pyris 1 calorimeter from PerkinElmer is an example of the use of the heat flux compensation method (power compensated). The other two, Mettler Toledo DSC 3 and Netzsch DSC 404 F1 Pegasus, are heat flux calorimeters with different heat flux measurement methods. On the one hand, the design differences affect the diversity of measurement procedures, but on the other hand, they create the possibility of highlighting a different parameter or property determined in the measurements.

In this case, a comparison was made of the capabilities of three selected scanning calorimeters in terms of their suitability for testing the specific heat of metals undergoing second-order phase transitions. The analysis covered the effectiveness and limitations of individual devices in determining the thermal characteristics of nickel (Ni) and iron (Fe), with particular emphasis on the temperature range, DSC signal resolution and repeatability of results.

2.1. PerkinElmer Pyris 1 power compensated DSC

PerkinElmer Pyris 1 DSC is a power compensated differential scanning calorimeter designed for thermal analyses and precise determination of the thermal properties of materials, in particular specific heat (c_p), in a temperature range up to 710°C. This limitation results from the use of resistive temperature sensors and compensating resistance heaters in the measuring head to implement the idea of power compensation. The apparatus enables quantitative determination of specific heat based on the analysis of the heat flow signal recorded during controlled heating of the sample relative to a reference material with known thermal parameters according to a so-called three-curve method [12]. This method involves using data from three test series – a baseline series, a series using a standard, and a series using the test sample – to determine the specific heat based on the equation:

$$c_{p,\mathrm{sp}}(T) = c_{p,\mathrm{ref}}(T)\,\frac{m_{\mathrm{ref}}}{m}\,\frac{f_{\mathrm{sp}}(T)-f_{\mathrm{base}}(T)}{f_{\mathrm{ref}}(T)-f_{\mathrm{base}}(T)}, \qquad (1)$$

where: sp − indicates a specimen, ref – reference, and base – baseline data.

The function f represents thermograms signal data that could be recorded in mW in the case of an enthalpy calibrated instrument or in arbitrary units in the case of an only temperature calibrated apparatus. Typical measurements are performed on samples weighing from 5 mg to 100 mg, placed in standard aluminium encapsulated crucibles with lids. A single crystal of sapphire (Al_2O_3), characterised by a well-defined c_p value, is usually used as a reference standard. The mass of both the test sample and the standard is determined with a resolution of 0.01 mg and recorded in the Pyris software.

The measurement is carried out in a protective atmosphere, most often purified nitrogen, with a controlled carrier gas flow

in the range of 20–50 ml/min. The standard heating rate is 5–20 K/min, but it is possible to use non-standard temperature profiles, including procedures with isothermal stops, which allow the elimination of dynamic effects such as the thermal inertia of the system.

The calorimeter's measuring range covers temperatures from room temperature to approximately 600°C, depending on the configuration of the apparatus and the crucibles used. As part of extended testing procedures, it is possible to use a cyclic heating and cooling regime with short isotherms between temperature steps. This approach allows for:

- improved accuracy of the determined c_p values,
- reduction of the impact of irreversible phenomena and thermal instabilities,
- independent analysis of the DSC signal in heating and cooling cycles, which increases the reliability and repeatability of the results.

The specific heat values are calculated using dedicated Pyris software. The programme compares the heat flow signals from the sample and the standard, taking into account their masses, and allows direct determination of c_p as a function of temperature. The data obtained are presented in the form of graphs and tabular summaries of $c_p(T)$ values, which can be exported for further analysis.

Baseline correction and signal smoothing functions are available as part of the data analysis. In the case of materials undergoing phase transitions, it is also possible to determine the characteristic transition temperatures and calculate the enthalpy of heat transitions.

2.2. Mettler Toledo DSC 3

The Mettler Toledo DSC 3 differential calorimeter is an advanced heat-flux DSC device used, among other things, for the precise determination of the thermal properties of materials, in particular specific heat capacity (c_p) as a function of temperature. The measurement is based on the analysis of the difference in heat flow signals recorded during the controlled heating of the sample and a reference standard with known thermal properties. The samples for analysis usually weigh from a few to several milligrams and are placed in standard aluminium crucibles, usually with a lid, without a seal. A sapphire disc (Al_2O_3) is usually used as a reference standard, as its thermal properties are well known and documented. Both the sample and the standard are weighed accurately (with a resolution of 0.01 mg and an accuracy depending on the type of balance) and then placed in two identical measuring chambers of the calorimeter. The test procedure data are as follows:

- the measurement is carried out in a controlled protective atmosphere, usually in a stream of nitrogen or argon, with a fixed gas flow,
- the standard heating rate is 10 K/min, although it can be adjusted to the specific characteristics of the material being tested,
- the measurement range covers temperatures from room temperature to approximately 600–700°C, depending on the configuration of the apparatus and the crucibles used.

The device determines the difference in heat flow between the sample and the reference standard as a function of temperature. Based on this data, the dedicated STARe software calculates the direct value of the sample's specific heat, taking into account the previously performed calorimetric calibrations. The results are presented in the form of a $c_p(T)$ graph and in tabular form, taking into account measurement uncertainty. The software allows baseline correction to be applied to compensate for any DSC signal drift. The results obtained can be compared with literature data or used for further analysis, such as calculating the enthalpy of phase transitions, analysing the thermal properties of composites, or modelling thermal processes in engineering systems.

2.3. Netzsch DSC 404 F1 Pegasus

The Netzsch DSC 404 F1 Pegasus differential scanning calorimeter is a high-temperature heat-flux DSC device designed for precise study of the thermal properties of substances, including specific heat capacity (c_p) determination, in a wide temperature range – up to 1600°C. The apparatus allows the use of the three-run method (the three-curve method presented in paragraph 2.1 [12]), which is the standard procedure for determining c_p in relation to a sapphire standard.

The analysis is carried out on samples weighing typically from a few to several milligrams, placed in cylindrical crucibles made of a suitable material. Platinum-rhodium (Pt/Rh), aluminium oxide (Al_2O_3) or graphite crucibles are used for specific heat measurements, depending on the expected temperature range and chemical properties of the sample material. A certified sapphire disc (Al_2O_3) with a well-defined specific heat, supplied by the manufacturer (Netzsch), is used as a standard. The mass of all measuring elements is determined with a resolution of 0.01 mg and entered into the Proteus® Thermal Analysis software.

Determination of specific heat using the three-measurement method involves the following steps:

- baseline measurement – recording the thermal background of the system with empty crucibles in the sample (S) and reference (R) positions,
- standard test – recording the reference thermogram for the standard material sample, most often sapphire – crystalline aluminium trioxide, in crucible S and reference crucible R,
- sample testing – measurement with the tested material in crucible S and the reference crucible in position R, which allows the recording of a thermogram that forms the basis for determining the specific heat capacity of the tested material.

All three measurements must be carried out under identical conditions, which means:

- using the same measuring vessels – crucibles,
- maintaining the same heating rate (e.g., 10 K/min),
- ensuring an identical protective atmosphere (usually nitrogen or argon) and the same volume flow rate, typically 50−70 ml/min,

- application of the same temperature change programme typically defined in the range from −170°C or 20°C to 1000°C or 1600°C, depending on the system equipment.

Specific heat calculations are performed by Proteus® Thermal Analysis software, which uses signal differences obtained from three measurements (baseline, reference, sample) applying appropriate calorimetric relationships. The results are presented in the form of $c_p(T)$ graphs and tables, with the possibility of further data processing: smoothing, mathematical model fitting and export to external analytical formats. The software enables the assessment of the repeatability and precision of measurements, as well as advanced thermal analysis of materials undergoing phase transitions, including the determination of transition enthalpy and characteristic temperatures.

The system is particularly suitable for testing construction materials, ceramics, metal alloys and functional materials used at elevated temperatures.

2.4. Subject of research

Given the methodological objective of the study, it was decided to attempt to determine the specific heat of pure elements for which reliable literature data on specific heat are available – samples of pure metals: nickel (Ni) and iron (Fe) [13]. These materials were selected not only due to their well-documented thermo-physical properties, including specific heat, but also with regard to a second-order phase transition within the temperature range available in standard DSC calorimetry. This allows for the comparison of experimental results with literature data and the assessment of the accuracy and sensitivity of the measurement methods used. The following metals were therefore selected for the study:

- Nickel (Ni) – the material used had a purity of ≥ 99.9%. Nickel is a metal undergoing a second-order phase transition – ferromagnetic-paramagnetic ordering – at a Curie temperature ranging from approximately 355°C (628 K) [14], through 357°C (630 K) [15], 358°C (631 K) [16,17] to 360°C (633 K) [13] (Fig. 1). Due to its relatively sharp peak, this transition is particularly useful as a reference signal in DSC measurements. The presented reference positions of the Curie point (T_C) of nickel, both in terms of the abscissa, i.e. the temperature T_C, as shown – both along the x-axis (i.e., the temperature) and the y-axis (i.e., the corresponding specific heat value) – define the range to which the position of this point, as determined in these experiments, should be referenced. In fact, almost all T_C position test results fall within the range of 355°C ≤ T_C ≤ 360°C.
- Iron (Fe): the iron used was also characterised by high purity (≥ 99.5%). In the analysed temperature range, Fe undergoes a ferromagnetic-paramagnetic phase transition with a Curie temperature close to 770°C (1043 K) [13,18]. Due to the temperature limitations of some devices, full parameterisation of this transformation was only possible using the Netzsch DSC 404 F1 Pegasus high-temperature device. However, the measurement range of the tests performed with this device also covered the first order $\alpha-\gamma$ phase transformation.

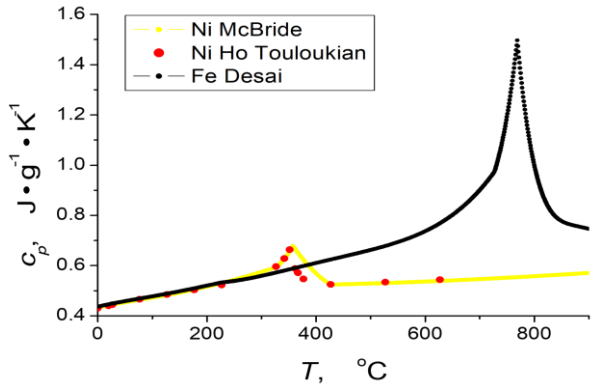


Fig. 1. Dependence of specific heat on temperature, showing the position of the Curie point as the peak of the second-order phase transition – measurements of specific heat for Ni [13,17] and Fe [18].

The nickel and iron samples were taken from a larger sample used for precise measurements of thermal expansion as a material with established thermal properties [19]. They were designed to ensure that the largest possible surface area of the bottom makes good contact with the bottom of the sample crucibles. The sample weights were selected to ensure compliance with the instruments' sensitivity requirements. All samples used in comparative tests are shown in Fig. 2.



Fig. 2. View of the samples used in the comparative tests.

Prior to measurement, the materials were subjected to thermal stabilisation (preliminary annealing) in a protective atmosphere in order to remove residual moisture and surface contaminants and to reduce the effects of history (e.g. stresses or oxidation). The temperature change programmes used are presented later in this paper.

3. Experimental research

3.1. Research methodology

The experiments using the three different differential calorimeters mentioned above were conducted using two temperature-change programs, which were compared in this study. The first program, developed for the PerkinElmer Pyris 1 DSC, utilised heating and cooling rate of 20 K/min applied in the temperature range from –40°C to 570°C in the range of 570−600°C, the rate was changed to 10 K/min in order to ensure stable conditions of

transition between the heating and cooling phases, in accordance with the dynamic characteristics of the apparatus (Fig. 3).

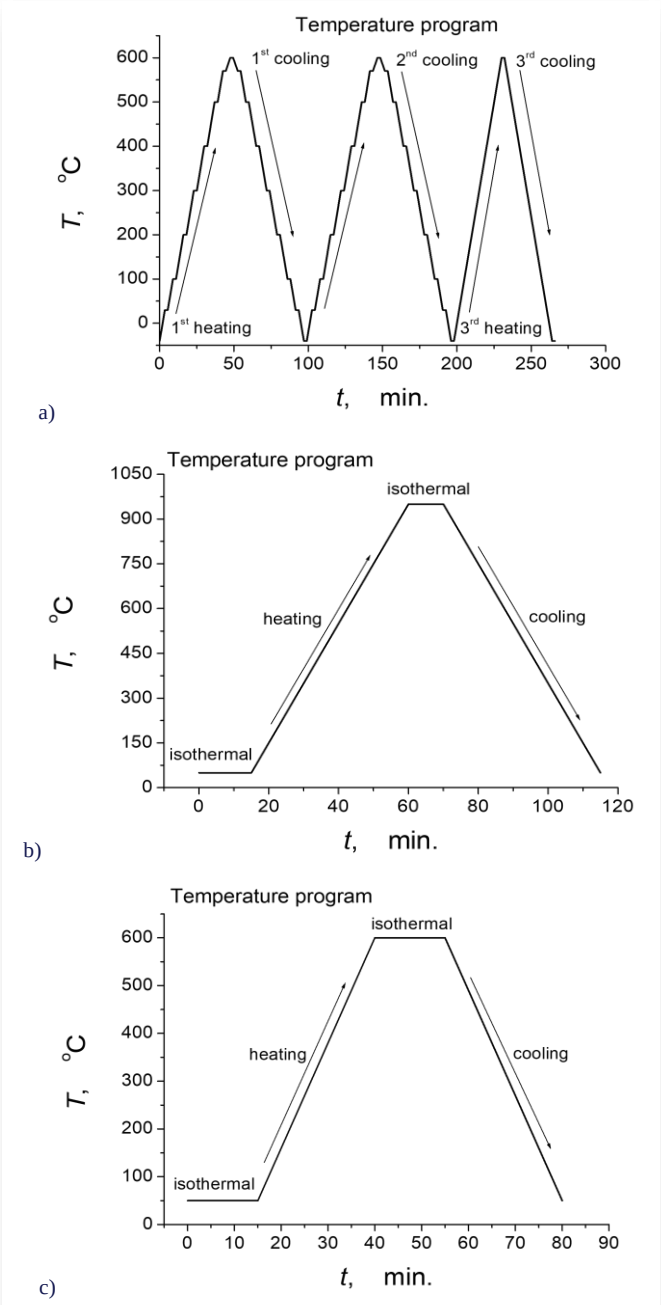


Fig. 3. Sample temperature programme for testing Ni (a, b) and Fe (c) materials on individual devices.

The temperature programme included two minutes of isotherm at the end of each heating and cooling stage in order to stabilise the temperature conditions and limit the impact of instrument equilibration time. The use of isotherms is an author's modification of the measurement procedure, aimed at improving the accuracy of determining the specific heat at constant pressure c_p [20,21]. The same program was used to conduct part of the research using the DSC 3 Mettler Toledo instrument. The measurements were carried out in a protective argon atmosphere at a constant flow rate of 20 ml/min (PerkinElmer) and in a protective nitrogen atmosphere at a constant flow rate of 150 ml/min (DSC 3 Mettler Toledo). The study also employed a standard heating and cooling program with a single ramp of linear temperature changes at a rate of 20 K/min in the range from room temperature to 600 °C (Ni: DSC F1 Pegasus 404 Netzsch and DSC 3 Mettler Toledo) or to 950°C (Fe: DSC F1 Pegasus 404 Netzsch; Fig. 3). When taking measurements with the Pegasus instrument, an argon flow rate was set to 70 ml/min. Data analysis was based on a standard calibration procedure using the so-called three-curve method and, additionally, the direct method (Mettler Toledo) including measurements for the reference system (empty aluminium vessels), reference material (sapphire) and the tested Ni and Fe samples. In accordance with the adopted methodology, the final results were obtained on the basis of DSC signals from the second or third heating and cooling cycle, i.e. after reaching a steady state in the sample-vessel-atmosphere system, as well as for the entire temperature range in order to present the discrepancies between individual temperature cycles.

3.2. Specific heat determination results

For calorimetric tests using DSC aimed at determining specific heat at constant pressure, two samples of Fe: with a mass of 230.72 mg in the shape of a cylinder with a diameter of approximately 4 mm (DSC 3 Mettler-Toledo tests), and an irregular in shape of 56.48 mg (PerkinElmer Pyris 1 and Netzsch DSC 4 F1 Pegasus studies) were applied. Two studied Ni samples were irregular in shape. The masses were equal to 63.90 mg (DSC 3) and 53.77 mg (Pyris 1 and DSC 4 F1 Pegasus). In both material cases, the investigated specimens were separated from a larger fragment of iron and nickel samples, respectively. It should be mentioned that the use of irregularly shaped samples is acceptable in DSC analysis, provided that the influence of the shape on the measurement uncertainty is taken into account and remains within the acceptable range of this uncertainty [22]. In the case of investigation of bulk metallic samples, these effects are negligible.

First, the temperature-dependent specific heat of the Fe sample, as determined in the experiments, will be analysed. The results are shown in Fig. 4.

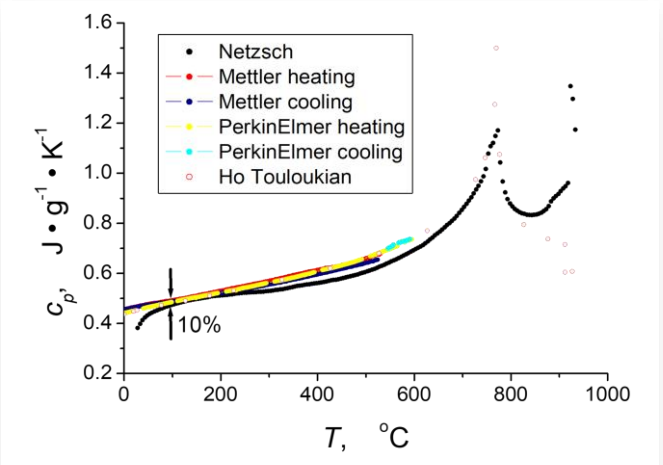


Fig. 4. Temperature characteristics of the specific heat of iron (Fe) determined using three different calorimeters, compared with reference data [13].

In addition to the measurement data collected within a temperature range limited to 600°C, the figure shows the results of measurements conducted using a DSC F1 Pegasus 404 calorimeter at temperatures up to about 950°C. When analysing these data, it is worth noting the significant deviation of the c_p values from those reported in the literature at temperatures above 850°C, which may indicate equipment limitations in this temperature range. In the ferromagnetic-paramagnetic transition region (T_C = 770°C [13]), the data consistency is more than satisfactory. Only this device made it possible to obtain data within the temperature range in discussion. For comparison, the results obtained using other calorimeters (Pyris 1, DSC 3) in the range up to 600°C are also presented, which show good agreement with the reference values.

For a more detailed analysis of the measurement results, Fig. 5 shows the variation of specific heat as a function of temperature over a narrower temperature range. The graph shows a high degree of consistency between the results obtained using Pyris 1 and DSC 3 calorimeters and the reference data. The data obtained using the F1 Pegasus 404 apparatus show a systematic underestimation of values compared to the other measurements. The results presented come from the third heating and cooling cycle, i.e. after the measurement conditions in the sample-vessel-atmosphere system had stabilised.

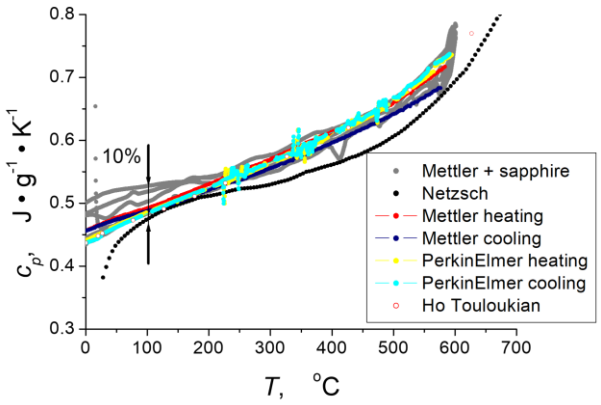


Fig. 5. The temperature characteristics of the specific heat of iron (Fe), determined using three different calorimeters, compared with reference data available in the literature [13].

The results of comparative studies on the specific heat of nickel as a function of temperature are shown in Fig. 6. The graph shows the results of measurements taken using three different calorimeters in the temperature range up to 600°C. It is important to note the significant deviation of the c_p value from the literature data in the case of measurements obtained using the DSC 3 calorimeter with the direct calculation method. The reason for this effect is difficult to identify, but the result itself illustrates the scale of problems that can be encountered in experimental studies of specific heat even for such well-defined materials as metallic elements.

Greater consistency between our test results and the data in the literature was achieved by using the three-curve procedure, referred to in the instrument's user manual as the "sapphire reference". This is illustrated by the comparison of the results of both types of measurements shown in Fig. 7. It should be noted

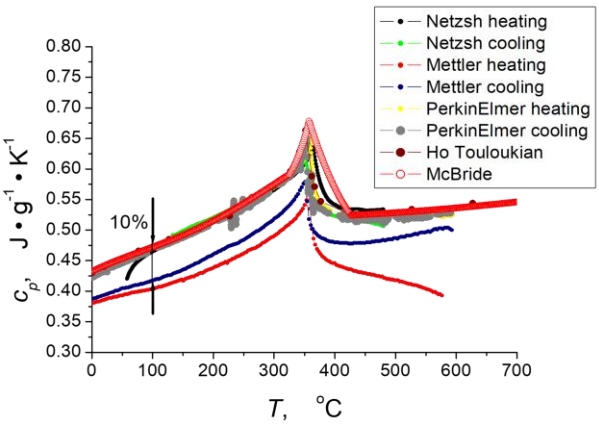


Fig. 6. The temperature characteristics of the specific heat of nickel (Ni), determined using three different calorimeters, compared with reference data available in the literature [13].

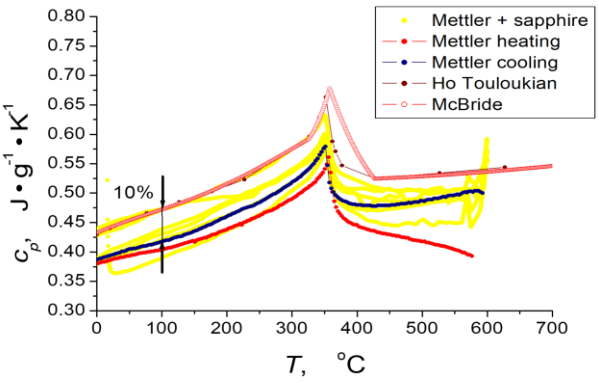


Fig. 7. Temperature characteristics of the specific heat of nickel (Ni) determined using a DSC 3 Mettler Toledo calorimeter by the three-curve method in comparison with reference data [13,18].

that the graph presents raw experimental data, which exhibit an artefact caused by irregular heating regime heat transfer in the form of apparent peaks at the extremes of the temperature range.

Based on the results of the specific heat measurements of nickel, the Curie temperature can be determined (T_C = 352°C [13]). The ferromagnetic transition is manifested by a characteristic lambda-shaped peak. The results of determining the position of the maximum of the temperature dependence of specific heat for the test results obtained using Pyris 1 and F1 Pegasus 404 devices are shown in Fig. 8a, and for the DSC 3 device in Fig. 8b.

The raw test results were further processed using a special computational procedure for approximation with B-spline curves [23] for cases where the range of the approximated data contained discontinuities in the first derivative of the temperature characteristic c_p. The test data for iron up to 600°C were approximated using a third-degree polynomial. This allowed us to determine representative specific heat characteristics as a function of temperature for each individual measurement. At the same time, the characteristic features of the peaks of the ferromagnetic phase transition were preserved. The results of the approximation, providing a summary illustration of the presented results in the context of their possible discrepancies, are shown in Fig. 9.

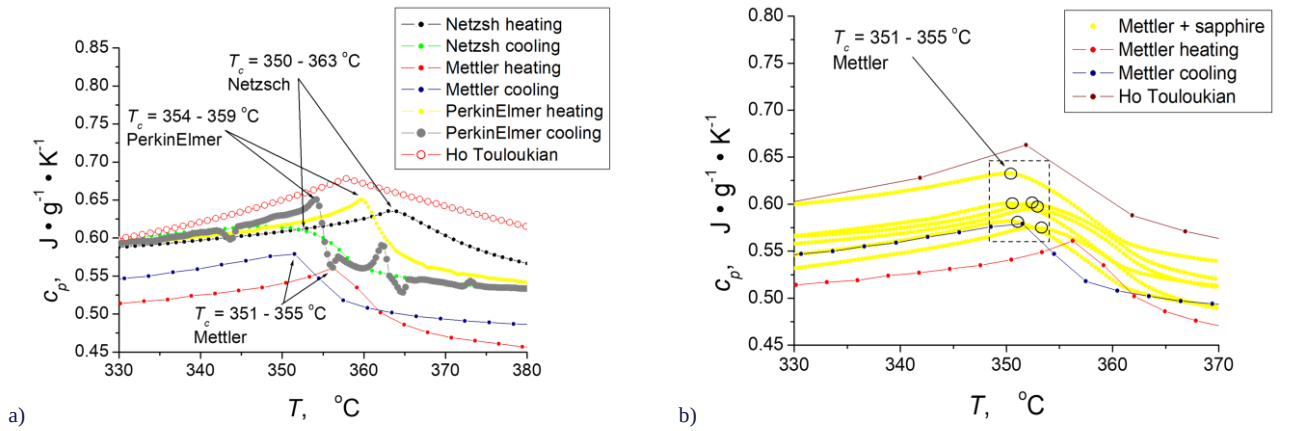


Fig. 8. Determination of the Curie temperature T_C for nickel: a) from measurements taken with Pyris 1 and DSC 404 F1 Pegasus devices, b) from calorimetric tests performed with a Mettler Toledo DSC 3 calorimeter.

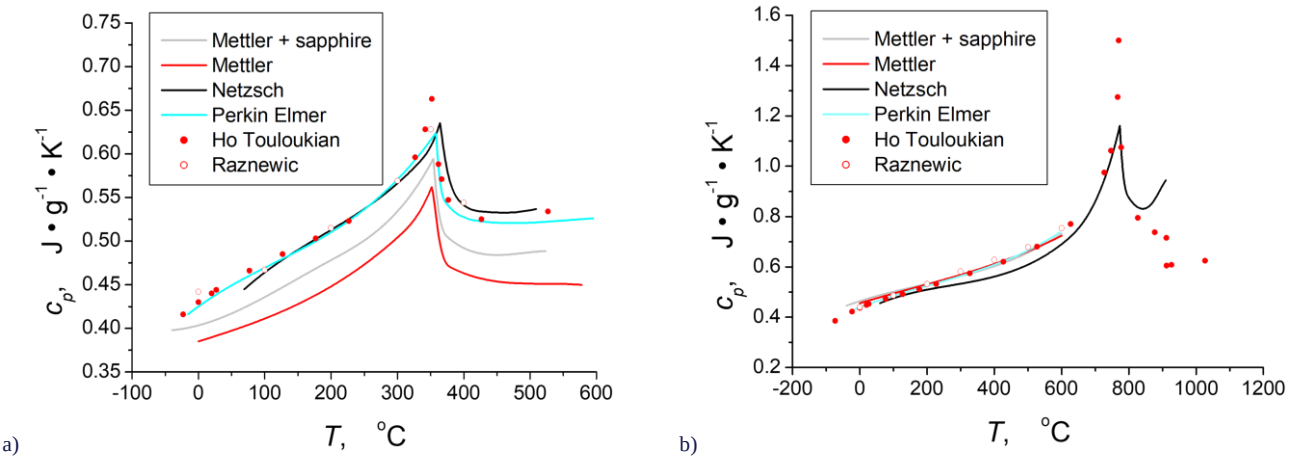


Fig. 9. Temperature-dependent specific heat at constant pressure characteristics for nickel (a) and iron (b) – B-spline [23] approximation results.

At the same time, these data provide an indirect answer to the question of measurement uncertainty. In thermal analysis studies, the emphasis is placed on the reproducibility of results and, where applicable, on agreement with data from reference materials [11]. It is possible to achieve better agreement than that shown in the data presented here. The results discussed above were obtained using standard procedures. Repeating measurements multiple times, using several samples, statistical averaging, and other such measures will yield a more satisfactory final result. However, this requires more effort and time. Therefore, the actual needs should be taken into account in each individual case.

It is also possible to estimate measurement uncertainty more accurately without resorting to interlaboratory comparisons. The problem, however, is that the necessary calculations must be performed for each individual case. And this time, it is not about, for example, the individual data of the tested samples, but rather about a set of data hidden within the calibration parameters. And when, for example, temperature calibration is involved, accounting for drift would require checking the calibration data each time. A case where temperature calibration data is critical is that of high-temperature-resolution measurements in the vicinity of phase transitions, as characterised in the monograph [20] and publication [24].

4. Conclusions

The aim of the study was to compare the metrological capabilities of selected methods and devices for determining the specific heat of materials undergoing second-order phase transitions, with particular emphasis on nickel and iron. The data obtained using three different differential scanning calorimeters (DSC), using both standard procedures and proprietary measurement methods, were analysed. In the course of the experiments, the influence of the test procedures used and the declared temperature change programmes on the specific heat values obtained and the results of the Curie point location identification were assessed. Particular attention was paid to comparing the results with available literature data and analysing the course of phase transitions. Based on the results obtained, the following conclusions were drawn:

- The tests showed that it is possible to obtain reliable specific heat measurements using the DSC method, even with irregularly shaped samples, provided that the measurement

conditions and uncertainty analysis are repeated. This is important from the point of view of industrial applications, where it is not always possible to prepare samples that comply with standard requirements.

- A comparison of three independent calorimeters (PerkinElmer Pyris 1, Mettler Toledo DSC 3 and Netzsch DSC 404 F1 Pegasus) allowed for an assessment of their metrological capabilities and the impact of device design and conversion algorithms on data quality. The Pyris 1 device ensures the highest repeatability and accuracy of results; the DSC 404 F1 Pegasus device allows satisfactory measurement results to be obtained in the high temperature range, while the DSC 3 device, despite the observed differences in results compared to the literature data, proved to be a device ensuring repeatable results of Curie point identification.
- The introduction of a proprietary measurement procedure with isothermal stops in the Pyris 1 apparatus made it possible to obtain consistent results in both heating and cooling regimes. This procedure can be a useful tool in further studies of phase transition materials, increasing the amount of available reference data.
- Analysis of the $c_p(T)$ data allowed the determination of characteristic parameters of the second type of phase transition for nickel. The convergence of the peak maximum temperature and the end of the transition in various measurements confirms the correctness of the methods used. This, of course, applies to the repeatability and accuracy of the measurement results that can be obtained.
- For iron, reliable mapping of the phase transition curve was only possible when using the DSC 404 F1 Pegasus apparatus. Slightly larger deviations in the low-temperature test results obtained with this device confirm the need for complementary tests.
- The specific heat values obtained were in satisfactory agreement with the literature data in the range up to 600°C. The test results obtained with the DSC 404 F1 Pegasus device show large differences from the reference data in the range above 850°C. Considering the difficulties in accurately determining the temperature characteristics of all thermo-physical quantities in the vicinity of phase transitions, especially of the first kind [19−21], this effect cannot be treated as a basis for questioning the correctness of the apparatus or the correctness of the measurement procedures. On the contrary, it is necessary to consider continuing this type of research using comprehensive complementary testing procedures. This will allow data to be obtained in the form of temperature dependencies of other thermophysical quantities [24], which will enable accurate identification of the issue and determination of reliable reference data.

The results of this research contribute to the methodology of calorimetry, providing both comparative data and practical guidelines for interpreting and verifying measurement results for metallic materials undergoing phase transitions. The results of the single-sample tests presented in this study provide detailed information regarding the selection of measurement procedures. This applies to the measurement procedure and the selection of the method for determining specific heat. Indirectly, they also provide guidance on the possible need to expand the project – by including a larger number of samples and cycles – in order to achieve statistical representativeness.

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