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Improvements in Data Reduction in Direct Pulse Heating Calorimetry[1]

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This paper describes improvements introduced in data reduction in direct heating pulse specific heat experiments. In calculations of specific heat, it is necessary to calculate the first derivative of the recorded temperature data as a function of time. The error induced by different numerical differentiation techniques can represent a significant part of the overall measurement error. Thus, different digital filtering techniques, differentiation, and smoothing algorithms were applied and tested to examine their influence on the minimization of errors induced by noise, which is unavoidable in measured signals. A minimum square error criterion was applied in designing digital filters, with arbitrary prescribed magnitude characteristics. Attention was paid to applications when one or more structural phase transitions in the specimen material occur within the temperature range covered by the experiment. The cases where the frequency spectrum of induced noise overlaps with the spectrum of temperature transient signals originating from phase transitions were analyzed in detail. The effectiveness of the methods of extracting the final specific heat data from a noisy signal using different digital filtering techniques is demonstrated.

KEY WORDS: data reduction; digital filters; hemispherical total emittance; noise filtering; pulse calorimetry; phase transitions; specific heat.

1. INTRODUCTION

A variant of the direct pulse heating calorimetry method was developed at the Institute of Nuclear Sciences Vinča in order to enable fast, productive, and reliable measurements of the specific heat of solid conductors in the range of contact thermometry. The method is used for measuring specific heat, electrical resistivity, and hemispherical total emittance of electrical

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conductors. Thermocouples, as temperature sensors, enable measurements from room temperature up to 2500 K.

Dealing with low-sensitivity thermocouples as is the high-temperature W–Re thermocouple combination, particular attention has to be paid to errors induced by different numerical techniques applied in data reduction. Differentiation, which constitutes an important part of the process of obtaining specific heat and hemispherical total emittance data, makes this error significant. The differentiation process amplifies induced noise, especially when the noise frequency spectrum overlaps with the signal spectrum. The aim of this paper is to analyze and improve data reduction methods in pulse heating calorimetry in order to reduce overall measurement uncertainty, particularly those used in computation of specific heat and hemispherical total emittance.

2. METHOD

The method is based on fast Joule heating of a specimen in the form of a wire to some predetermined high temperature. Data on the specimen current (I), the voltage drop (U) across the measurement zone, and the emf of the thermocouple (E) located at the center of the effective sample measurement zone, are recorded during heating and the initial part of the cooling period. Specific heat is obtained from the relation

$$C_p = \frac{UI - \varepsilon\sigma A(T^4 - T_a^4)}{m(dT/dt)_h} \tag{1}$$

where T is the temperature of the specimen, t is time, m is the mass of the effective specimen, T_a is the ambient temperature, ε is the hemispherical total emittance, A is the effective surface area, and σ is the Stefan–Boltzmann constant. Hemispherical total emittance at a specific temperature T is calculated from

$$\varepsilon = \frac{UI}{\sigma A(T^4 - T_a^4)[1 - (dT/dt)_h/(dT/dt)_c]} \tag{2}$$

The subscripts h and c in the above equations refer to the heating and cooling period respectively. Electrical resistivity ρ is determined from the electrical resistance of the specimen as

$$\rho = \frac{U}{I}\frac{S}{l_e} \tag{3}$$

where S is the specimen cross-sectional area and l_e is the effective sample length between the potential leads. Description of the measurement system and evaluation of the method are given in Ref. 1.

3. DATA REDUCTION

In the computation of specific heat, electrical resistivity, and hemispherical total emittance, direct application of Eqs. (1)–(3) to the raw experimental data can lead to erroneous results, particularly for C_p and ε, which involve the very noise-sensitive calculations of the first derivative of temperature vs. time function. To overcome these problems, some sort of a preliminary reduction must be performed. For this purpose, several different techniques, such as selective digital filtering and digital differentiation methods, combined with other data reduction techniques, were tested.

For satisfactory reconstruction of the original signal from collected discrete samples, which always limit the acquired signal frequency band, the sampling frequency of data acquisition must be properly related to the bandwidth of the original signal. The general criterion for an adequate sampling process states that the minimum sampling frequency has to be at least twice the bandwidth of the measured signal. For a true data reproduction, common practical criterion requires that the sampling frequency should be at least 10 times the bandwidth of the analog signal. The problems which arise in measuring C_p, ρ, and ε using the pulse technique are that the frequency bandwidth of the signals varies with the heating rate and the duration of the pulse. Particular difficulties appear if the specimen material is subjected to structural phase transformations within the tested temperature range, which causes fast changes in material properties and affects the measured signals in the higher-frequency band.

In practice, a superimposed noise in measurement signals has magnitude which decreases slow toward the higher frequencies. Due to signal sampling, aliasing folds the noise spectrum, so the noise present in the sampled data has a rather flat contribution. Furthermore, sometimes other particular shapes of the noise spectrum might be present (such as line frequency voltage at 50 Hz), so before assuming that the noise spectrum is flat, the detailed spectrum analysis has to be performed. Precise resolution of the signal spectrum from the noise spectrum in the presence of material structural phase transitions is essential for preserving changes of the material properties.

4. DIFFERENTIATION FILTERING METHODS

The differentiation operation is essential in deriving specific heat and hemispherical total emittance from the raw experimental data. Therefore, for experimental data reduction, the design of an optimal wideband digital differentiator is of utmost importance.

Of the two types of digital differentiators, the nonrecursive type consists of coefficients that multiply the samples of the acquired signal as a discrete convolution. The recursive digital differentiator type filters the input discretized signal in the same way, except that a discrete convolution of previously computed values is added. Moreover, in contrast to the nonrecursive type, recursive differentiators can be designed to have usable bandwidths up to 100% of the whole frequency band, but with a relatively low order. Consequently, for differentiation of experimental data on temperature for obtaining specific heat and hemispherical total emittance, recursive digital differentiators were chosen as most suitable.

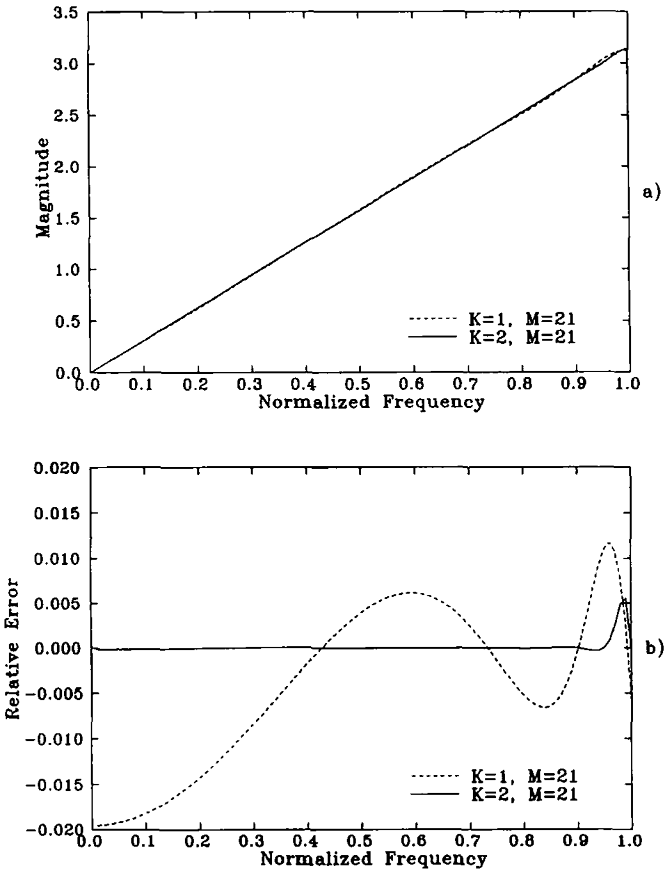

Fig. 1. (a) Magnitude characteristics of one-section ($K=1$) and two-section ($K=2$) recursive differentiators; (b) relative magnitude error of one-section ($K=1$) and two-section ($K=2$) recursive differentiators.

Table I. Coefficients of One-Section and Two-Section Recursive Differentiator

$K=1$, $M=21$, $A=0.36637364$		$K=2$, $M=21$, $A=0.36792357$	
a1	−0.32917379	a1	0.82070953
b1	−0.67082621	b1	0.13088158
c1	0.85938969	c1	0.95235296
d1	0.10210105	d1	0.04814614
		a2	−0.10609814
		b2	−0.89390186
		c2	0.94155350
		d2	0.19434558

The digital differentiating process has an inherent difficulty consisting in the impossibility to meet simultaneously two opposing demands. On the one hand, preservation and reconstruction of minute details contained in the acquired data require saving information contained within the higher-frequency range. On the other hand, superimposed noise, with a flat contribution in the spectrum, will be amplified in the differentiation process, especially at higher frequencies. This usually causes problems in differentiating the temperature transient signal of a material having structural phase transitions, as this depart of the temperature signal spectrum lies in the higher-frequency range. So application of a recursive differentiator with a wide usable bandwidth, combined with a delicate subsequent lowpass filtering, was the optimal choice.

The magnitude characteristic of an ideal differentiate increases linearly up to the normalized frequency of 1.0 (Nyquist frequency), representing half of the sampling rate. The phase is $\pi/2$ radian for frequencies up to the Nyquist frequency. Design of differentiating recursive filter applied in this case was based on the criterion of minimization of the square error of the magnitude frequency response [2–4]. The mathematical description of a recursive differentiator is

$$Y_n = \sum_{k=0}^{k=K} C_k X_{n-k} + \sum_{k=1}^{K} D_k Y_{n-k} \tag{4}$$

or, in canonical form, the transfer function is

$$H(z) = \pi A \prod_{k=1}^{k=K} \frac{1 + a_k z^{-1} + b_k z^{-2}}{1 + c_k z^{-1} + d_k z^{-2}} = \pi A \prod_{k=1}^{K} \frac{(1 - z^{-1} a_{i1})(1 - z^{-1} a_{i2})}{(1 - z^{-1} b_{i1})(1 - z^{-1} b_{i2})} \tag{5}$$

with each cascade of K section containing two zeros and two poles. If the desired frequency response is a response of the ideal differentiator with transfer function $H_d(e^{j\omega}) = j\omega$ at a discrete set of frequencies $\{\omega_i\}$, $i = 1, 2, \ldots, M$, the sum of square error in the frequency domain is

$$E = \sum_{i=1}^{M} [|H(e^{j\omega_i})| - |H_d(e^{j\omega_i})|]^2 \tag{6}$$

The problem is to find such values of all unknown coefficients in Eq. (5) that will minimize E in Eq. (6). This is a nonlinear problem, and an iterative method, as, for instance, the Marquardt method, must be used.

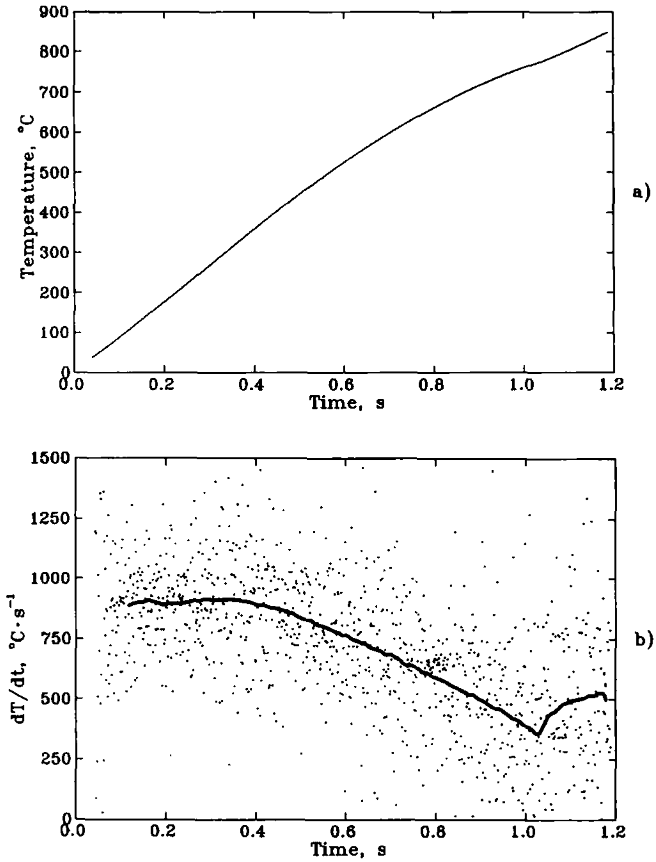

Fig. 2. (a) Temperature signal vs time function; (b) Nonfiltered and filtered first derivative of temperature signal vs time function.

When this method is applied to one-section ($K = 1$) and two-section ($K = 2$) differentiators, coefficients will converge to the values shown in Table I.

Figure 1 shows the magnitude characteristics and relative magnitude errors for both one-section and the two-section differentiator. It should be noted that the magnitude error for a two-section filter is below 10^{-3} for about 95% of the bandwidth, which is sufficiently accurate for the calculation of the first derivative of a transient temperature signal.

In order to locate the frequency limit from which the signal can be separated from the noise, it is necessary to compute the power spectrum

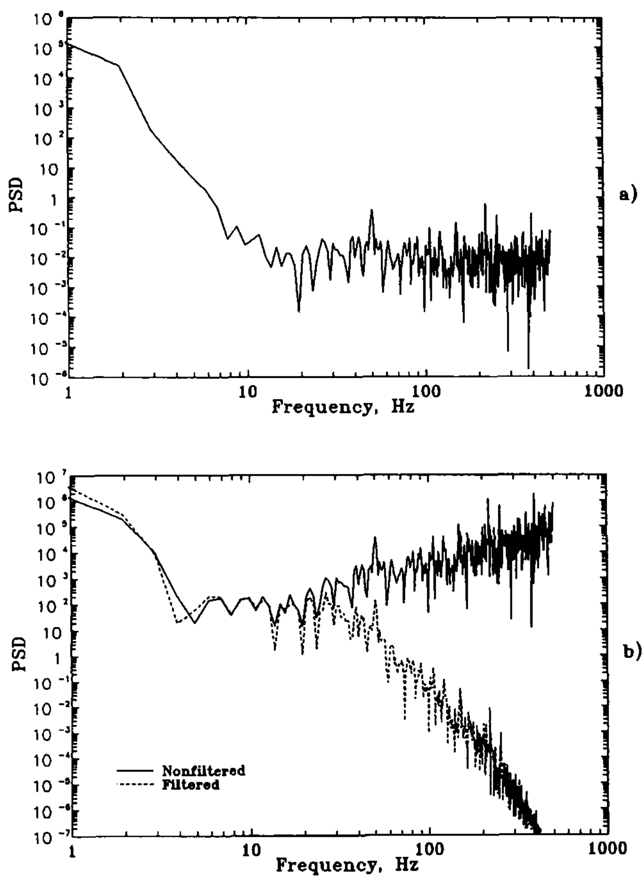

Fig. 3. (a) Power spectrum density of temperature signal; (b) power spectrum density of nonfiltered and filtered first derivative temperature signal.

density (PSD) of the input signal. Figures 2a and 3a show the transient temperature signal in a pulse heating experiment applied to electrolytic iron (NIST SRM[4] 1463) and the PSD of the temperature signal taken at a 1-kHz sampling rate. The PSD shows a spectrum with useful information in the low-frequency range and confirms that the noise tends to be flat across the whole spectrum. Differentiation of the temperature signal applying the above-mentioned recursive differentiator ($K=2$, $M=21$), gives very noisy results for the first derivative (Fig. 2b), but also all minute details contained in the differentiated signal are saved with the relative error shown in Fig. 1b ($K=2$, $M=21$). Due to the magnitude characteristic of the two-section differentiator, moving from low- to higher-frequency PSD of the raw first derivative of the temperature signal increases (Fig. 3b). Application of this method ensures that all frequencies are differentiated with sufficient accuracy.

5. LOW-PASS DIGITAL FILTERING

For elimination of the high-frequency noise and for allocation of the material structural phase transitions in the first derivative of the temperature signal, the Butterworth low-pass filter could be used, as its magnitude characteristic is maximally flat in both pass-band and stop-band ranges. The short transition band can be achieved using a higher order of the transfer function. Figure 2b shows nonfiltered and filtered first derivatives of the temperature signal, with a cutoff frequency of 25 Hz. The PSD of these filtered signals are shown in Fig. 3b.

Since they are not symmetric, Butterworth and most other recursive filters have a phase shift between input and output data, which varies with frequency. This is particularly important in filtering the first derivative of the transient temperature signal in the case when the precise temperature of material structural phase transitions has to be detected. To eliminate this phase shift, it is necessary to repeat the same filtering process in the reverse direction. If there is a phase shift at a given frequency in the first pass through the filter, there will be the same phase shift of the opposite sign at the same frequency in the second pass. So processing of the output result in both directions eliminate the phase shift. Variants of this method also allow elimination of the phase shift resulting from differentiation process.

Combining filtered first derivatives of the temperature signal, with the voltage and the current data, according to Eq. (1), the specific heat is obtained; the results are presented in Fig. 4a.

It is evident that low-pass filtering did not eliminate all the noise present in the specific heat data. When the frequency spectrum of superimposed

[4] U.S. National Institute of Standards and Technology, standard reference material.

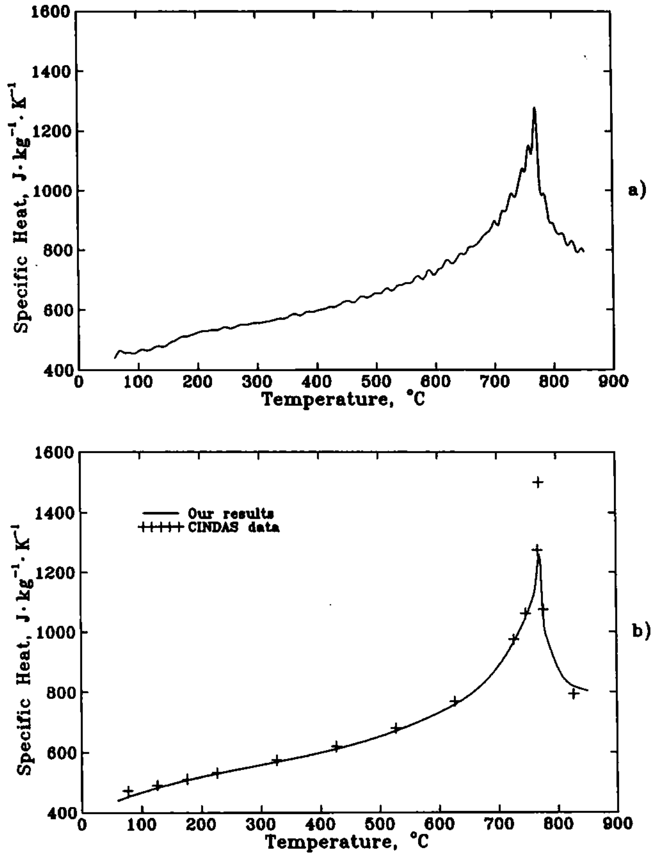

Fig. 4. (a) Single-experiment specific heat values of iron; (b) averaged specific heat values of iron.

noise overlaps the spectrum of the temperature signal, especially within the structural phase transitions, conventional filtering techniques are not sufficiently effective. To eliminate such low-frequency noise, after applying the low-pass filtering mentioned above, repeating the experiments and multiple specific heat data averaging appear to be suitable technique, provided that the following two conditions are met:

(a) obtained experimental data must be repeatable from experiment to experiment, i.e., specimen properties must not be subjected to irreversible changes; and

(b) low-frequency superimposed noise must be of a random nature.

After running 10 or more experiments, and summing the collected specific heat data, random low-frequency noise fluctuations in the final data set will usually almost vanish, and the signal-to-noise ratio will increase proportionally to the square root of the number of averaged experiments. Final specific heat data obtained following the above procedure is shown in Fig. 4b, in addition to the CINDAS[5] recommended values [5].

6. CONCLUSIONS

Summarizing the items discussed in this paper, several conclusions can be derived. For preserving most of the original measured quantities over a wide frequency range, a sufficiently high sampling rate must be chosen. This is particularly important for postexperimental reconstruction of the acquired signals. Computation of the power spectrum density is important for its analysis, and removal of superimposed noise from significant experimental data.

Due to the fact that the computation of specific heat and hemispherical total emittance is directly related to the computation of the derivative of the temperature signal, it is clear that the application of a very accurate wideband differentiator is necessary. For this purpose, recursive differentiators were designed with a usable bandwidth of 100% of the full band. Further data reduction included separation of high-frequency noise observed in the PSD analysis, using a Butterworth low-pass filter with a narrow transition band.

The precise location of the temperature of material phase transformations requires delicate applications of the mentioned filtering techniques, due to a possible signal phase shift. A two-way filtering technique was applied to eliminate this problem.

To produce final thermophysical property values, superposition of several (10 to 20) repeatable experiments was performed, which leads to minimization of the remaining low-frequency noise.

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