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Ten Years of Parameter Estimation Applied to Dynamic Thermophysical Property Measurements[1]

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The parameter estimation theory is considered the best way to estimate thermophysical properties from dynamic experiments. This approach deals with measurement and model errors in a statistical context and provides useful information to optimize the experiment. The experience gained in ten years of implementation of inverse algorithms based on the parameter estimation theory (OLS, MAP, and Kalman filtering) is summarized and presented. Several examples of estimation of thermophysical properties using transient and pulse techniques are reported and discussed. The thermal conductivity, specific heat capacity, and total hemispherical emissivity of different materials (light insulators, Pyrex, and niobium) are presented and compared with data obtained with consolidated techniques and with literature data.

KEY WORDS: dynamic methods; light insulators; niobium; parameter estimation theory; Pyrex; thermophysical properties.

1. INTRODUCTION

In recent years, the interest in dynamic techniques for thermophysical property measurements has rapidly grown in many research laboratories. Several portable apparatus, based on transient methods, are also commercially available. Dynamic methods are attractive because experiments are reasonably short in duration and contain a great deal of information which can be properly used to investigate the thermal behavior of the material. Moreover, from a philosophical point of view, the dynamic thermal regime better adheres to the normal state of materials because in nature "everything moves." But dynamic techniques require more complex modeling of

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the physical phenomenon and more sophisticated signal processing. As the estimation process is usually based on some inverse solution (analytical or numerical), the unavoidable presence of errors in the measured data may have a detrimental effect in the final estimates, because of the ill-posed nature of any inverse problem. For this reason, besides a great precision in the measurement technique, the key to a precise and reliable estimation of thermophysical properties in dynamic regimes is the choice and implementation of the estimator algorithm. First of all, measured data should be analyzed in a statistical context in order to estimate not only the thermophysical properties but also the related variances, which are as important as the unknown properties. Moreover, to optimize the estimation process as a whole, the design of the dynamic experiment and of the estimation algorithm should proceed in an integrated way and continuous improvements in both need to be made. The parameter estimation theory offers a tool to satisfy, in a comprehensive and flexible way, all the above needs and should be considered the best approach for these applications.

In this work the experience gained in ten years of implementation of inverse statistical algorithms OLS (ordinary least squares), MAP (maximum *a posteriori*), and Kalman filtering, is summarized and presented. Several examples of thermophysical property estimation using transient and pulse techniques are reported and discussed pointing out the advantages of the parameter estimation approach with respect to more traditional methods. The thermal conductivity, specific heat capacity, and total hemispherical emissivity of different materials such as light insulators, Pyrex and niobium, are presented and compared with measurements performed with consolidated techniques and with literature data.

2. STRATEGY FOR THERMOPHYSICAL PROPERTY ESTIMATION

Thermophysical properties measured by dynamic techniques can be evaluated following two different strategies summarized in the block diagram of Fig. 1. A simple and common way consists of the use of the deterministic approach shown in Fig. 1a. This method requires the adoption of some simple and invertible analytical solutions for a dynamic heat conduction problem. Then, an experimental apparatus able to reproduce the dynamic experiment as close as possible to the assumptions made in the theoretical formulation is designed. The thermal response of the specimen is measured, and the thermophysical properties of the material are reconstructed by introducing, in the inverse formula, data affected by errors (noise and biases) that are generally unknown. This simple and immediate method does not provide any information about the entity of errors affecting the final estimates. Undesirable biases can originate not only from the typical measurement errors affecting the signals of temperature probes, heat flux transducers, etc., but also from the difference between the physical model (governing equations and boundary condition) idealized in the theoretical formulation and the real experiment. In some cases, by performing a separate series of simulations more representative of the real experimental situation, it is possible to quantify partially the effect of model errors by means of correction factors. However, there is no assurance that the distortion of the estimated parameters can be fully compensated by the correction factors because these are evaluated in an open loop and separately from the effective experiment. In addition, further undiscovered errors may affect the input data with unpredictable consequences.

A better solution is to follow the parameter estimation approach shown schematically in Fig. 1b. The inverse solution of the heat conduction equation is formulated on a statistical basis and obtained by numerical methods. Hence, there is no need to select dynamic experiments with simple boundary conditions whose analytical solution must be known and invertible. Furthermore, a preliminary analysis can be developed to maximize the sensitivity coefficients of the unknown parameters and to design a near optimum experiment [1]. This means that thermophysical properties can be estimated with the greatest precision compatible with practical restrictions due to experimental constraints. For example, the optimal initial and boundary conditions can be selected in order to assure minimum variances for the estimated parameters [2], or the number and location of thermal sensors can be optimized [3]. Of course, placing one or more sensors in a region where the sensitivity coefficients are extremely high implies that very important information can be acquired during the dynamic experiment but, at the same time, the influence of measurement errors and biases is also maximized. As a consequence, a compromise on a “near optimum”experiment must often be accepted.

The statistical formulation of the inverse problem permits every kind of signals affected by noise to be processed and identification of both thermophysical properties and their associated variances. These values, together with temperature and heat flux residuals, are essential indices to check the accuracy and the reliability of the entire reconstruction process. Many different measurement and process mismatches can be detected and compensated by including, in the inverse solution, further additional models. For example, the knowledge of the location where thermal sensors are placed inside the specimen can be identified with great accuracy [3, 4]. Errors in temperature measurements by contact probes in a transient regime can be adequately compensated [5, 6]. The uncertainty of sensor calibration on the variances of the estimated thermophysical properties can be included in

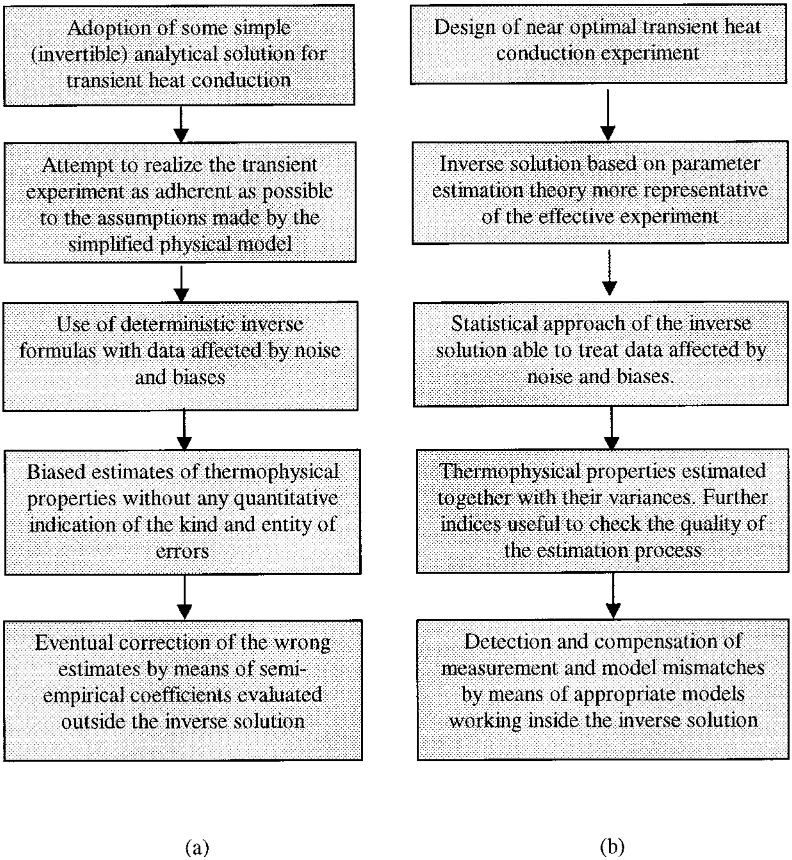

Fig. 1. Estimation processes of thermophysical properties by dynamic techniques: deterministic method (a), parameter estimation approach (b).

the inverse conduction problem [7, 8]. The bias compensation may be optimized by identifying, in the same experiment, both the thermophysical properties and the unknown coefficients appearing in the additional models. In fact, the unknown correction factors are identified by considering the effective arrangement of the instrumented specimen in the real experimental situation. From the above considerations, it is evident that the parameter estimation approach is a correct strategy that offers many advantages.

Based on the parameter estimation theory, different algorithms were implemented for application to thermophysical property estimation from dynamic experiments: the standard OLS or MAP techniques [3, 9] and more sophisticated stochastic approaches like the Kalman filtering [2, 4, 10–12]. A detailed description of the above algorithms is beyond the scope of the present work, and the interested reader should refer to specialized literature. A complete development of the OLS and MAP estimators is available in Ref. [1], while the Kalman filtering is treated in several books [13–16]. This work is devoted to some examples of the application of the Kalman filter to the reconstruction of thermophysical properties for different materials in various temperature ranges, making reference to experiments realized at both the Dipartimento di Termoenergetica (DITEC, University of Genova, Italy) and the CNR Istituto di Metrologia "G. Colonnetti" (IMGC, Torino, Italy).

3. EXAMPLES OF THERMOPHYSICAL PROPERTY ESTIMATION

3.1. Transient Experiment on a Light Insulator

The first example refers to the simultaneous identification of the temperature-dependent thermal conductivity and specific heat capacity of a light insulator in a single experiment. Complete details of the experimental apparatus developed at the DITEC are reported in Refs. [9, 11]. The specimen, shown schematically in Fig. 2, is placed between heating and cooling plates, and the transient experiment is realized by supplying electric power to the ribbon resistance inside the heating plates, and/or by varying the temperature of the cooling plates with the circulation of a thermostatted fluid. In a typical experiment at atmospheric pressure, the temperature increase can be of the order of 150 to 200 K per hour, while the temperature of the hot plates can be reduced to the initial temperature in a few minutes by means of the cooling plates. The heating plates, the specimen, and the cooling plates are placed inside an air-tight, thermostatted bell-shaped vessel to control both the pressure and the microclimate of the gas surrounding the specimen. Figure 2 shows also a typical

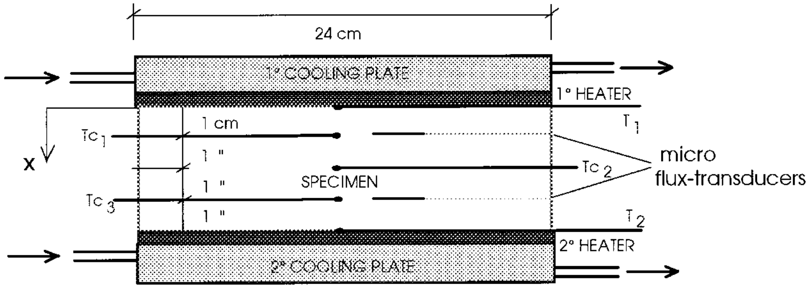

Fig. 2. Schematic diagram of the transient experiment on light insulating materials.

set of instrumentation. Thermocouples T_1 and T_2 are inserted in proper grooves of the heaters to measure the temperature imposed (or resulting) during the transient on the two opposite boundaries of the specimen. Other thermocouples (Tc_1, Tc_2, and Tc_3) are inserted at various and known distances inside the material to measure the temperature response of the specimen. The transient heat flux is measured by micro-foil heat flux transducers placed between adjacent layers of material at known positions. Starting from an initial and uniform temperature, normally room temperature, the desired thermal transient is imposed and, from the measured response of the specimen in temperature and heat flux, the thermal conductivity and specific heat capacity are simultaneously reconstructed as a function of temperature by solving the associated inverse nonlinear heat conduction problem. An example of the simultaneous identification of the temperature-dependent thermal conductivity and specific heat capacity (continuous lines) is shown in Fig. 3. The dashed lines are the associated 99% confidence regions predicted by the estimator. The measured material is an EPB (expanded perlite board) insulator, in rigid panels. It is an open-pore light insulator, with an apparent density of about 149 $kg \cdot m^{-3}$.

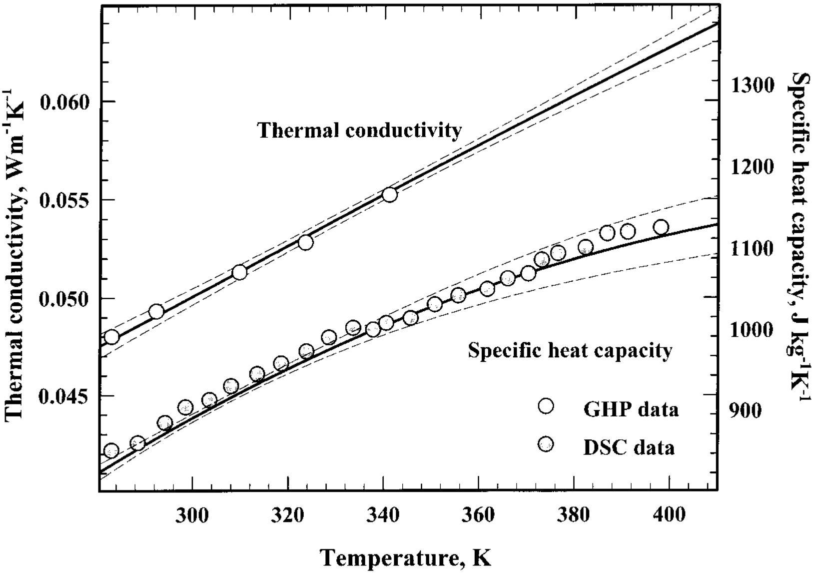

Fig. 3. Temperature-dependent thermal conductivity and specific heat of EPB (expanded perlite board) simultaneously estimated from a single transient experiment and compared with other measurement techniques: guarded hot plate (hollow circles), differential scanning calorimeter (full circles). The broken lines represent the 99% temperature-dependent confidence regions predicted by the estimator.

Practical experiences in transient measurements on this kind of material suggest the following considerations. Heat transfer by radiation and convection is negligible for EPB so that a pure conductive model is appropriate. The porous specimen must be accurately dried before the experiment because during heating any residual moisture causes sudden processes of evaporation, migration, and condensation. These phenomena cause large distortions of the temperature profiles with respect to the assumed pure conductive model, and the estimation process becomes biased. In general, the identification of the temperature-dependent specific heat capacity is more sensitive to the model bias induced by moisture than is the thermal conductivity. It is possible to find an optimal sequence of boundary conditions that can be used to minimize the variances of the reconstructed properties and to improve the robustness of the inverse solution for both thermophysical properties [2]. The great sensitivity of temperature profiles to the moisture evaporation and migration was used for implementing a more complete heat and mass diffusion problem in which simultaneous identification of thermal and vapor diffusion coefficients was achieved [17, 18]. The uncertainty in the knowledge of the real position of thermal sensors arranged inside the specimen was the major source of measurement errors. This was adequately compensated by including in the inverse solution the location of each inner sensor as a further unknown parameter to be estimated [3, 4]. The error due to the transient temperature measurements was compensated by adding appropriate thermal coupling models (measurement junction-material) in the inverse problem [5, 6]. Finally, the noise of the measurement signal was minimized by executing a large number of temperature measurements using oversampling. At the end of the iterative identification process, standard deviations of temperature residuals of the order of 6 to 7 mK were obtained with a total temperature excursion of about 150 K. Small confidence regions and unbiased temperature residuals are good indices of a correct and reliable estimation process. Figure 3 also reports results obtained with more consolidated techniques. Hollow circles refer to thermal conductivity data measured by the GHP (guarded hot plate) method, while full circles are specific heat capacity data measured by the DSC (differential scanning calorimeter) technique. Both thermal conductivity and specific heat capacity estimated in a single transient experiment comply very well with GHP and DSC results.

3.2. Transient Experiments on Pyrex

The second example refers to the simultaneous identification of the temperature-dependent thermal conductivity and specific heat capacity of Pyrex in a set of transient experiments based on the small perturbation of

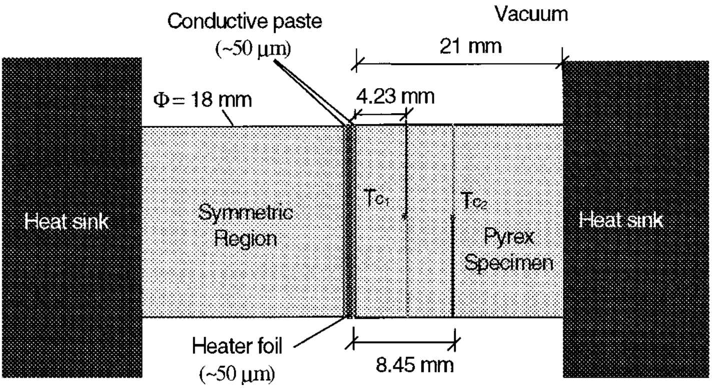

Fig. 4. Schematic diagram of the transient experiment on pyrex.

a specimen subject to a heat pulse [19]. These experiments are part of the development work at the IMGC of a new apparatus based on this principle (schematic diagram in Fig. 4). The different parts of the cylindrical specimen are all made with Pyrex CRM039, a certified reference material for thermal conductivity. The specimen assembly is housed in a vacuum enclosure immersed in a water thermostat. Sharp heat pulses with a typical duration of around 2 s are applied with the heater using different energy inputs, and the material response is monitored with thermocouples Tc_1 and Tc_2. The maximum temperature variations are of the order 1.8 to 3.6 K and occur shortly after the end of the current pulse, with the transient decaying in a few minutes. A total of 62 experiments were analyzed, at three different thermostat settings between 293 and 342 K, using different experimental conditions. The analyzed results are presented in Fig. 5, with the hollow circles and the full circles indicating the average thermal conductivity and specific heat capacity, respectively, obtained at each temperature. The bars represent the complete range of values in all experiments. The continuous line is the certified thermal conductivity value of CRM039 [20], along with its 95% confidence limits at 1.7%.

In this type of experiment, originally designed for simple deterministic inverse formulas, the form of the excitation function (energy pulse), with its sharp increase of heat flux and temperature on the boundary and inside the specimen, tends to emphasize all possible measurement and model errors in the first part of the experiment. Moreover, it is difficult to know the exact time at which the energy pulse starts and the total pulse duration. This may cause large temperature residuals at the beginning of the experiment. Conversely, because of the short duration of the input energy pulse, in the remaining part of the experiment the temperature gradient inside the

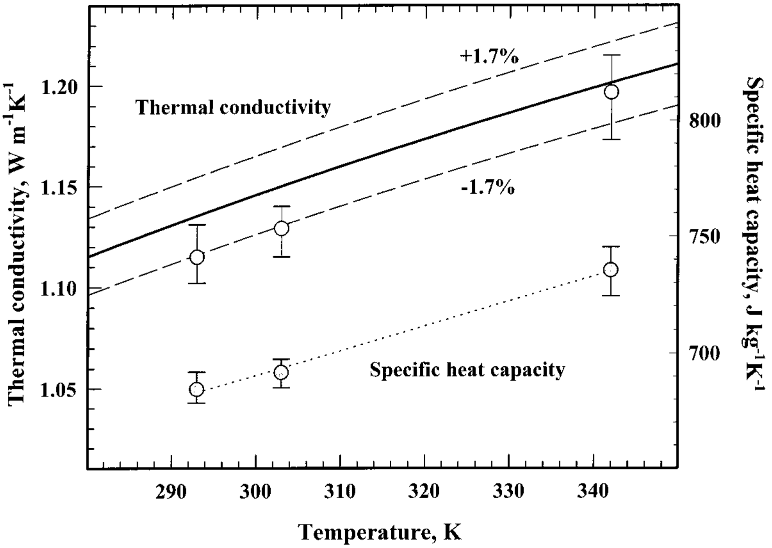

Fig. 5. Pyrex thermal conductivity (hollow circles) and specific heat capacity (full circles) estimated simultaneously in transient experiments with heating pulses. The solid line represents the certified thermal conductivity of the material with its 95% confidence regions.

specimen tends to decrease and, therefore, the information tends to fail. If we use statistical methods coupled to a numerical formulation of the heat conduction model, a more suitable boundary condition could be imposed on the hot surface of the specimen. The input heat flux should start with a soft increase of power in order to reduce the effect of the initial bias. At the same time the input energy should be maintained for a longer period of time in such a way as to preserve a high level of information in the remaining part of the experiment. Obviously the numerical formulation of the inverse heat conduction problem requires both a finite axial length of the specimen and a knowledge of some thermal boundary condition for the cold surface. To this aim a second thermocouple has been inserted in the specimen at a known position (details in Fig. 4). Other possible sources of bias are: the uncertainty in the knowledge of the position of the probe junctions inside the specimen, errors in the dynamic regime of the temperature probes, the finite thickness of the heater and of the conductive paste, and other minor effects due to heat losses by radiation through the lateral surfaces. All the above effects were modeled and included in the Kalman filter to improve the estimation quality. The present experimental results, although of a preliminary nature and obtained with sharp heat pulses, are in reasonable agreement with the certified values.

3.3. Pulse Experiments on Niobium

The last example refers to the simultaneous identification of the temperature-dependent specific heat capacity and hemispherical total emissivity of niobium over a wide temperature range (1050 to 2650 K).

The experimental data were obtained with the pulse heating apparatus of IMGC [21] using a tubular niobium specimen with a small rectangular blackbody hole (schematic diagram in Fig. 6). The specimen was self-heated to high temperatures with a current pulse of subsecond duration.

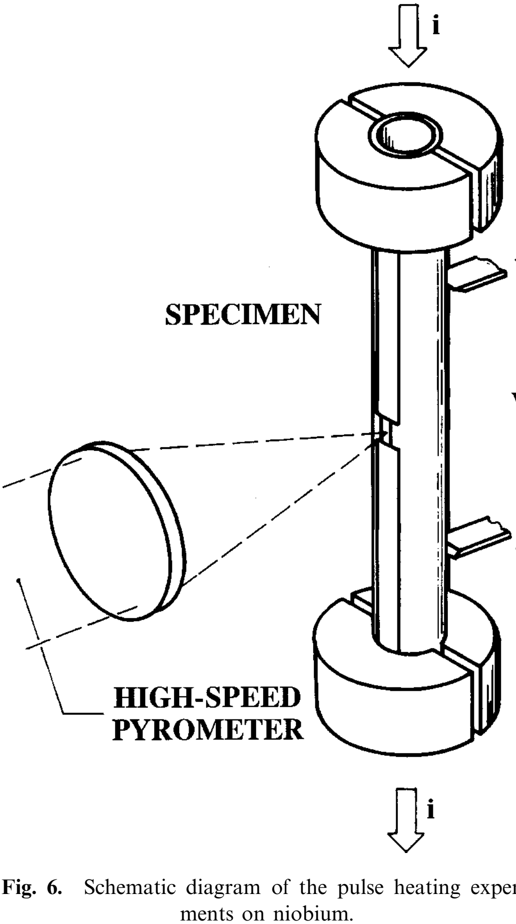

Fig. 6. Schematic diagram of the pulse heating experiments on niobium.

The current in the specimen, the voltage drop in the central part, and the blackbody temperature were measured with millisecond time resolution during heating and during the initial part of the free cooling period.

The analysis was performed on nine experiments reaching different high temperatures; earlier work using the same data had demonstrated that the Kalman filter and the classical method of elaboration provided final property results of comparable accuracy [22]. The present analysis was improved to include confidence limits for both thermophysical properties and to extend the identification of hemispherical total emissivity to the entire temperature range. The experiments are characterized by different current pulse intensities and duration and, therefore, different heating rates and maximum temperatures.

To optimize data acquisition, the measurement time step and the input gain of the pyrometer A/D converter unit are changed during the experiment with a different sequence in each experiment. Moreover, the quality (variance) of each temperature measurement is also variable for two reasons. The first is because the pyrometer noise depends on the level of the measured temperature, and the second is because the electronic noise superimposed to the temperature signal depends on the actual gain selected for the A/D converter. As a consequence the variance associated with each temperature measurement changes continuously and must be evaluated by considering the effective pyrometer transfer function and the sequence of adopted gains. In spite of this evident nonhomogeneity, all the experiments have been analyzed simultaneously by the Kalman filter. The sequence of the temperature signals coming from the various experiments was processed and weighed in an optimal way by taking into account the actual sensitivity and variance of each independent signal. Very low confidence regions for the estimated thermophysical properties were obtained.

The results of the analysis for the total hemispherical emissivity of niobium are presented in Fig. 7. Since this property is strongly dependent on surface conditions, a comparison with literature data has limited significance. The specific heat capacity results are compared in Fig. 8 with literature values from other pulse heating experiments in the same temperature range. As it may be expected, the closest agreement was obtained with the IMGC-1985 data [23] because these measurements were performed on four tubular specimens (different from the one used in this work) using similar experimental conditions. The agreement is remarkable in consideration that completely different sets of measurements and of specimens are involved, that measurements were performed several years apart and clearly with different calibrations, and that the IMGC-1985 data were obtained with a classical approach without use of the Kalman filter.

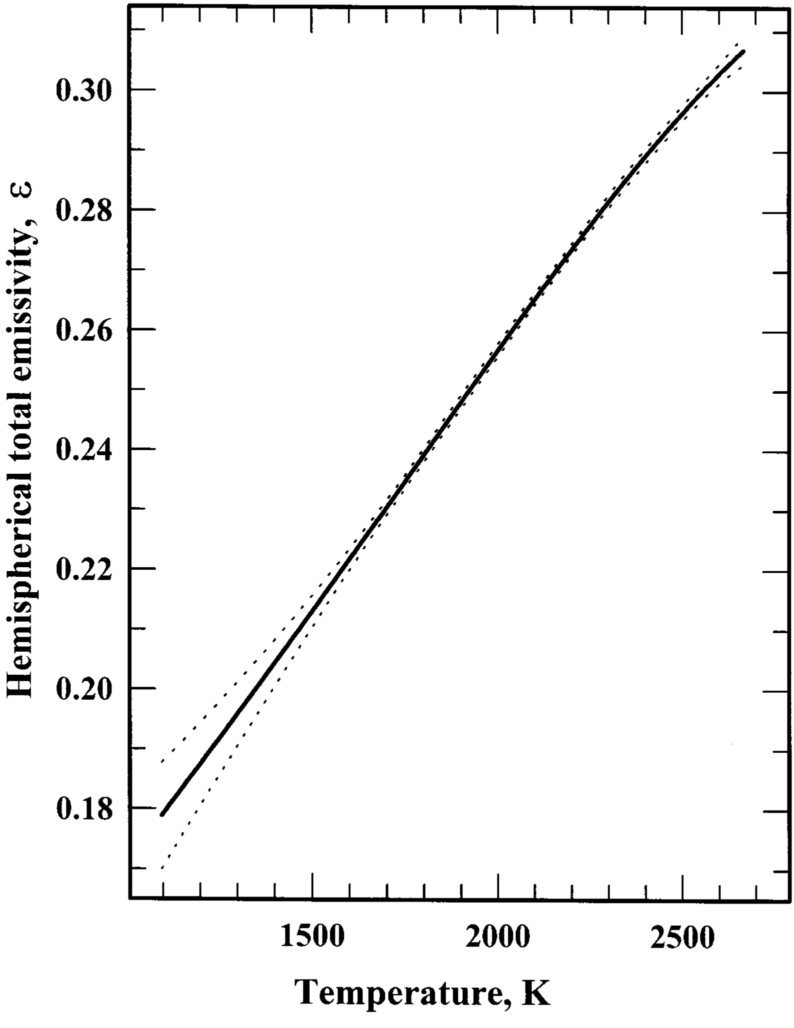

Fig. 7. Total hemispherical emissivity of niobium estimated by Kalman filtering applied to pulse heating experiments. The dotted lines indicate the 99% confidence regions predicted by the estimator.

The IMGC-1999 data are also in very good agreement with the present results in the temperature range above 1800 K, and show differences of 1 to 2% below 1800 K. Part of the difference may be due to the fact that the IMGC-1999 measurements [24] were performed on strips with the simultaneous determination of the normal spectral emissivity of the strip by integrating sphere reflectometry. The various sets of IMGC experimental

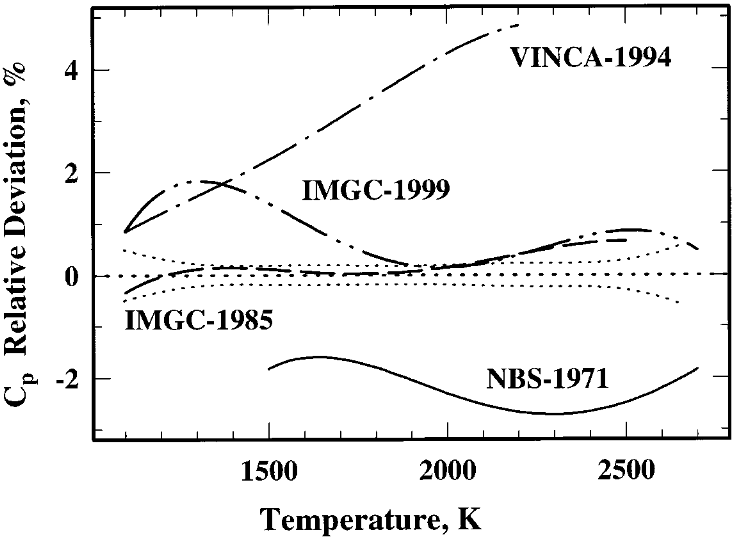

Fig. 8. Deviation plot of literature data from the specific heat capacity of niobium estimated by Kalman filtering applied to pulse heating experiments. The dotted lines indicate the 99% confidence regions predicted by the estimator. Literature references are identified in the text.

results have always indicated a difference of about 2% from similar measurements performed on tubular specimens with a blackbody hole at NBS in 1971 [25], and the difference is confirmed. The measurements VINCA-1994 [26] were obtained with a cylindrical specimen, and the temperature was measured by using spot welded thermocouples on the sample.

4. CONCLUSIONS

The use of statistical algorithms like the Kalman filtering for estimating thermophysical properties of materials from dynamic experiments can be considered as the most reliable and comprehensive approach. Many advantages can be achieved including the design of "near optimum" experiments and, therefore, the possibility to estimate thermophysical properties with the greatest precision compatible within the experimental constraints; several kinds of measurement and model errors can be processed and compensated. Several useful indices are available at the end of the identification process to assess the quality of the estimated properties. The above technique offers a powerful tool as parameter estimator and stimulates the design of more ambitious and intriguing dynamic experiments for evaluating thermophysical properties of materials.

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