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Thermal property characterization of fine fibers by the 3-omega technique

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HIGHLIGHTS

- An improved model for suspended wire 3 omega measurement.
- Quantification on the radiation induced measurement error.
- Numerical simulation validating the improved model.
- Sensitivity analysis to find measurement range minimizing uncertainty.

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ABSTRACT

The 3 omega method is one of few reliable measurement techniques for thermal characterization of micro to nanoscale suspended wires or fibers and has been applied for measurements of carbon nanotubes and silicon nanowires. However, the models described in the past were either complicated for analysis or simplified from a more complete solution. In addition, the past models cannot be implemented directly when using a more reliable measurement configuration with a Wheatstone bridge. In this work, a simpler, explicit model, is developed to describe the heat transfer process through a suspended wire for measurement of its thermal properties. Generic trends and values of the 3ω harmonic voltage amplitude and phase responses clearly indicate the frequency limits for thermal conductivity and heat capacity determination and ideal conditions for thermal diffusivity estimation. Based on a sensitivity analysis, these limits are confirmed and appropriate frequency ranges for thermal conductivity and diffusivity are recommended. Radiation influence on the measurement results is quantified and correlated to a dimensionless radiation parameter. Two methods are presented to determine sample thermal properties independent of lateral heat losses and validated by numerical experiments using COMSOL. Uncertainty analysis was also derived by Taylor series expansion with calculated parameter sensitivities.

1. Introduction

The commonly used configuration of the 3ω method is the heater-on-substrate system [1,2], in which a thin metal strip is evaporated on a sample surface, acting as both a heater and resistive thermometer. An alternating current (ac), at frequency ω, passes through the heater inducing a temperature change which propagates into the substrate at a frequency of 2ω. By monitoring the voltage response due to the change of resistance with temperature at the third harmonic frequency, the thermal conductivity can be determined. With an additional direct current (dc) offset, thermal property information can be obtained from 1ω or 2ω as well as detailed in Refs. [3,4]. However, as was concluded in Ref. [3], the 3ω measurement is generally the best choice for extracting thermal properties.

Several models are presented in the literature for temperature profiles in a suspended wire heated by ac current [4]. Many of these models were intended for modeling the thin wire probe used in scanning thermal microscopy (SThM) where the application is for thermal property measurement of a substrate contacting the probe apex. The 3ω method may also be applied to obtain the thermal properties of the suspended wire itself, as is detailed in this work.

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Though having similarities to the SThM probe model, for the suspended wire, the heat transfer model does not include the contact boundary condition required for the probe models. Thus, the probe solutions cannot be applied directly.

The 3ω method has been applied to micro- to nanoscale diameter fine fiber thermal conductivity and heat capacity determinations [5–8]. Lu et al. [6] modeled a suspended wire between two heat sinks arriving at a series solution using the impulse theorem. The final solution, used in other studies [8–10] for carbon nanotubes and silicon nanowire measurements, contains only the first term of the infinite series. Expressions for the thermal conductivity and heat capacity were derived from the voltage amplitude measurement because it contains all desired thermal property information; however, the more reliable phase response, which only provides thermal diffusivity was never used. Hou et al. [7] simulated the coupled suspended wire and substrate system and rendered a complicated solution including the wire-substrate interaction. As demonstrated in the analysis section, their model does not yield considerably different results from the current model considering the wire sample only. Modeling only the suspended wire removes the requirement for the unknown thermal contact resistance between sample and substrate, which alters the result significantly if inappropriately estimated.

Previously, the sample radiation influence on thermal property determination was either neglected, or only evaluated on a specific sample with no guiding conclusion reached [7]. In a previous study for the electrothermal measurement for fine fibers [11], the deviation of measured thermal conductivity and diffusivity from the reference values, caused by neglecting radiation influence, was related to a dimensionless radiation parameter. With this relationship, data reduction can be made using the simple model that neglects radiation, and then correcting for its influence in a final step.

The objective of this research is to provide a model for thermal characterization of suspended wires by the 3ω technique and to fully quantify its characteristics. It gives a complete and practical approach to measure thermal conductivity and heat capacity of a suspended wire. Radiation influence is evaluated in terms of the dimensionless radiation parameter. Some practical aspects regarding constant current and constant voltage sources are presented. A methodology is presented by which thermal properties may be obtained independent of sample length (lateral heat losses). Finally, a sensitivity analysis is presented for each measured thermal property.

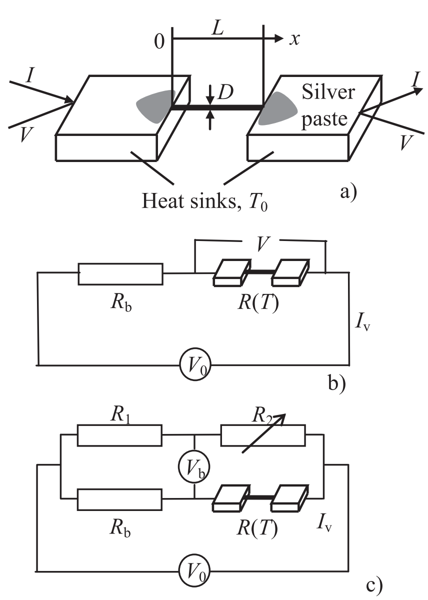

Fig. 1. Schematic of the 3ω measurement: a) current source connected directly to the sample, b) voltage source with a balance resistor R_b, and c) voltage source with a Wheatstone bridge.

2. Theoretical model

2.1. Constant current source

The fin-shaped governing equation presented in this work is similar to those in Refs. [4, 12] for Wollaston-wire probes used for SThM but with different boundary condition and application. Fig. 1a presents a schematic diagram of the sample setup for the 3ω method. The sample, with a large length (L [m]) to diameter (D [m]) aspect ratio is suspended between two highly thermally conductive heat sinks. A modulated electrical current is passed through the sample (electrically conductive or metal coated non-conductive fibers) causing a temperature rise (ΔT [K]) due to the Joule heating effect. The modulated current, I [A] and heat generation, q''' [W m^{-3}] can be expressed as

$$I = I_0 \cos \omega t, \tag{1}$$

$$q''' = I^2 R(T) \big/ V_s = I_0^2 (1 + \cos 2\varpi t) R(T) \big/ (2V_s) \tag{2}$$

where I_0 is the current amplitude [A], ω is the angular frequency [rad s^{-1}], $R(T)$ is the instantaneous resistance [Ω] of the sample at temperature T [K] and V_s is the volume [m^3] of the sample. The Joule heating contains constant and periodic components.

The sample resistance as a function of temperature change can be expressed as

$$R(T) = R_0 + R'\Delta T = R_0(1 + \alpha_T \Delta T) \tag{3}$$

where R_0 is the sample initial resistance [Ω], R' is the calibrated slope of temperature–resistance relationship [Ω K^{-1}] and α_T is temperature coefficient of resistivity [K^{-1}].

Even numbers of ω in Eq. (2) induce even number harmonics of temperature rise ΔT. With knowledge of the R', sample temperature rise can be monitored by its resistance change. With constant current, the resistance change is reflected by the monitored voltage [V] of the sample.

$$V_c = IR(T) = I_0 R_0 \cos \omega t + I_0 R' \cos \omega t \cdot \Delta T(2n\omega t), \quad n = 1, 2, \ldots \tag{4}$$

To use Eq. (4), the sample temperature change needs to be solved. Due to the large sample aspect ratio, the heat transfer process can be modeled by 1-dimensional (1D) heat conduction with lumped convection/radiation in the radial direction and a volumetric source/heat generation term. In terms of $\Delta T = T - T_0$ where T_0 [K] is the environment temperature, the governing equation can be written as

$$\frac{1}{\alpha}\frac{\partial \Delta T}{\partial t} = \frac{\partial^2 \Delta T}{\partial x^2} - \frac{4h_r}{Dk}\Delta T + \frac{I^2 R(T)}{kV_s} \tag{5}$$

where k is thermal conductivity [Wm^{-1} K^{-1}], α is diffusivity [m^2 s^{-1}], $h_r \approx 4T_0^3 \varepsilon \sigma$ is the linearized radiative heat transfer coefficient [Wm^{-2} K^{-1}] through the sample lateral surface, ε is the emissivity and $\sigma = 5.67 \times 10^{-8}$ Wm^{-2} K^{-4} is the Stefan–Boltzmann constant. The measurement should be conducted in high vacuum (<0.001 Pa) so that convective heat loss is negligible.