Temperature-dependent specific heat of suspended platinum nanofilms at 80–380 K*

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Metallic nanofilms are important components of nanoscale electronic circuits and nanoscale sensors. The accurate characterization of the thermophysical properties of nanofilms is very important for nanoscience and nanotechnology. Currently, there is very little specific heat data for metallic nanofilms, and the existing measurements indicate distinct differences according to the nanofilm size. The present work reports the specific heats of 40-nm-thick suspended platinum nanofilms at 80–380 K and $\sim 5\times 10^{-4}$ Pa using the 3ω method. Over 80–380 K, the specific heats of the Pt nanofilms range from 166–304 J/(kg·K), which are 1.65–2.60 times the bulk values, indicating significant size effects. These results are useful for both scientific research in nanoscale thermophysics and evaluating the transient thermal response of nanoscale devices.

Keywords: suspended platinum nanofilms, specific heat, size effect, 3ω method

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1. Introduction

Metallic nanofilms are important components of nanoscale electronic circuits, nanoscale sensors, and nano-electro-mechanical systems (NEMS). The accurate thermophysical characterization of metallic nanofilms is a key issue for both nanoscience and nanotechnology. Specifically, suspended metallic nanofilms have been used as resistive thermometers to probe nanoscale steady-state thermal and thermoelectric transport in the well-developed T-type method[1–4] and the H-type thermal bridge method.[5,6] In these methods, the in-plane electrical and thermal conductivities of the metallic nanofilm itself are prerequisites that are calibrated in advance using the direct current self-heating method.[7–9] In this electrical self-heating method,[7–9] the suspended metallic nanofilms are integrated with four probing electrodes, and a one-dimensional heat conduction model is used to effectively describe the direct current self-heating process, while the average temperature of the nanofilm is obtained from its own temperature-dependent resistance. The electrical and thermal conductivities of suspended metallic nanofilms have been measured many times,[1–9] and the results are distinctly different from the corresponding bulk values. The electrical and thermal conductivities of suspended 40-nm-thick platinum nanofilms were found to be less than half of the bulk values,[1–4,7–9] a fact that is explained by the surface and grain boundary scatterings.[8,9]

The specific heat of metallic nanofilms has seldom been investigated, although the specific heat is an important quantity when using metallic nanofilms to probe nanoscale transient thermal transport. In a theoretical study, Chen and Dames[10,11] used the low-dimensional Debye sub-model for elastic continuum boxes to simulate the temperature-dependent specific heat of low-dimensional materials. In this sub-model, the key parameters for determining the specific heat are the sound velocity, geometry, and the boundary conditions (fixed, free, or periodic). The results showed that the temperature-dependent specific heat follows a power law with respect to the temperature at low temperatures, where the power law exponent equals the dimensionality. Below 10 K, the specific heat of 1D materials is one order higher than that of 2D materials and two orders higher than that of 3D materials, whereas the specific heats of 1D, 2D, and 3D materials are nearly the same above 100 K. The simulation results were in accordance with the measured specific heats of TiO_2 nanotubes.[10] Experimentally, Yu *et al.*[12–14] developed a microscale calorimeter and measured the specific heats of 20–340-nm-thick Cu and 40–1150-nm-thick Al nanofilms supported on Si wafers in a vacuum. The results showed that the specific heats of Cu and Al nanofilms increase with decreasing thickness. At 300 K, the specific heat of the Cu nanofilm was 10% higher than the bulk value when the thickness was 100 nm and 65% higher when the thickness was

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20 nm, whereas the specific heat of the Al nanofilm was 42% higher than the bulk value when the thickness was 40 nm. Yu *et al.*[13,14] further simulated the specific heats of Cu and Al nanofilms with < 10 nm thickness, and attributed the enhancement in specific heat to the softening of phonons at the grain boundaries and the increased thermal expansion coefficient. Lugo *et al.*[15,16] measured the specific heats of Al, Au, and Cu nanofilms supported on glass at room temperature and atmospheric pressure based on the nanofilms' transient response to pulse currents, and reported that the specific heats of 20-nm-thick Al, Au, and Cu nanofilms were, respectively, 1.60, 1.78, and 1.58 times that of the corresponding bulk values, suggesting significant size effects. However, Queen and Hellman[17] reported no size effects on the specific heats of metallic nanofilms. They measured the specific heats of 52.5-nm-thick, 30.3-nm-thick Cu and 54.6-nm-thick Au nanofilms supported on SiN_x using a SiN_x-based nanoscale calorimeter, and obtained specific heats almost the same as the bulk values. Currently, there are still very few data for the specific heats of metallic nanofilms, and the existing measurements indicate variable size effects. Therefore, more measurements are required to evaluate the size effects of the specific heats of metallic nanofilms. In addition, the measured samples in previous studies were supported on Si, SiN_x, or glass substrates, and there is a lack of experimental data on the specific heats of suspended samples, which are widely used as nanoscale sensors. Moreover, the specific heats of suspended nanofilms can differ from those of supported ones because of surface interactions, and thus it is necessary to experimentally investigate the specific heats of suspended metallic nanofilms.

The 3ω measurement method[18–21] is effective for simultaneously measuring the thermal conductivity and specific heats of suspended wires, and has been widely applied in measuring the specific heat of microscale wires. In the 3ω method, an alternating current with 1ω frequency is used to heat the suspended sample, and the thermal conductivity and specific heat can be simultaneously extracted from the frequency-dependent 3ω signals. The present paper reports the measurement of specific heats of 40-nm-thick suspended platinum nanofilms at 80–380 K using the 3ω method.

2. Measurement principle

In the 3ω method, an alternating current with 1ω circular frequency is passed through a suspended nanofilm, and the electrical power varies at a frequency of 2ω. This results in a 2ω vibrating temperature rise in the nanofilm.[18–21] Because the electrical resistance of the Pt nanofilm varies linearly with temperature, the electrical resistance of the suspended nanofilm will also vary at 2ω and give rise to a 3ω voltage. The thermal conductivity and specific heat can be simultaneously extracted from the relationship between the 3ω voltage and the frequency.[18–21] As for the heat conduction model, because the length of the nanofilm is much larger than the width and thickness, the heat conduction process in the suspended Pt nanofilm can be described by a 1D transient diffusive equation with a volumetric heat source, and the 3ω voltage can be derived from the average temperature rise of the suspended nanofilm. When the input current is $I = I_1 \sin(\omega t)$, the 3ω voltage of the suspended Pt nanofilm, $V_{3\omega}$, is expressed by[19]

$$V_{3\omega} = \frac{1}{2} R' R_0 I_{1,\mathrm{rms}}^3 \mathrm{abs}\,(Z)\,, \tag{1}$$

where R' is the slope of the resistance–temperature curve, i.e., $R' = \mathrm{d}R/\mathrm{d}T$; R_0 is the electrical resistance of the Pt nanofilm at ambient temperature; $I_{1,\mathrm{rms}}$ is the root mean square of the input 1ω current; abs(Z) is the modulus of the complex number Z, which is expressed by[19]

$$Z = \frac{[\sinh(\beta) - \sin(\beta)] + \mathrm{i}\,[\sinh(\beta) + \sin(\beta) - \beta\,(\cosh(\beta) + \cos(\beta))]}{2\beta\rho c_\mathrm{p} V \omega\,[\cos(\beta) + \cosh(\beta)]}, \tag{2}$$

where $\rho = 21450$ kg/m^3 is the density of Pt;[18–21] c_p is the specific heat of Pt nanofilm at constant pressure; V is the volume of the Pt nanofilm; ω is the circular frequency of the input current (equal to 2π times the frequency f); and β is a function of the thermal diffusivity expressed by

$$\beta = \sqrt{\omega \rho c_\mathrm{p} L^2 / \lambda}, \tag{3}$$

where L and λ are the length and the thermal conductivity of the Pt nanofilm, respectively. According to Eqs. (1)–(3), the relationship between the 3ω voltage and the current frequency is determined by the specific heat and thermal conductivity of the test nanofilm. These two thermophysical properties can be simultaneously obtained by fitting the $V_{3\omega}$–f curve.

In practice, it is more common and convenient to use a voltage source instead of a current source. In this case, the resistance fluctuation of the sample should be much less than the total resistance in the circuit to guarantee the current source assumption applied in the above formulas.

3. Experimental

3.1. Sample fabrication

Suspended platinum nanofilms were fabricated via a series of nanofabrication processes, mainly consisting of elec-

tron beam lithography (EBL), electron-beam-induced physical vapor deposition (EB-PVD, Showa Shinku SEC-12), and etching processes on the Si wafer. First, a 320-nm-thick EBL resist, ZEP520-A, was spin coated on a 1 cm × 1 cm Si wafer with 260-nm-thick SiO_2, and a pattern with four electrodes and a narrow ribbon was developed in the EBL resist. Second, 8-nm-thick Ti and ∼ 40-nm-thick Pt layers were successively deposited on the Si wafer by EB-PVD, with the Ti layer used as adhesive material between the Pt and the Si wafer. The electron beam voltage was 8 kV and the purities of the Ti and Pt were 99.99% and 99.98%, respectively. The EBL resist and the metal on the resist were then removed during the liftoff process, leaving the metallic nanofilm with the electrode–nanowire patterns on the Si wafer. Finally, the narrow Pt nanofilm was suspended by etching the Ti and SiO_2 underneath the nanowire using 37% buffered HF and further etching the Si via CF_4 plasma. The total etching depth was about 1 μm. Figure 1 shows an SEM image of the suspended nanofilm at a tilted angle. These nanofabrication processes and the sample geometry were exactly the same as those used in our previous measurements of the thermal conductivity of metallic nanofilms.[1–4,7–9] In the present work, three ∼ 40-nm-thick suspended Pt nanofilms were fabricated in three different fabrication runs. The thicknesses of the Pt and Ti layers were successively measured by a profilometer (BRUKER Dektak XT) and the widths and lengths were read from the top-view SEM images. These measurements are listed in Table 1.

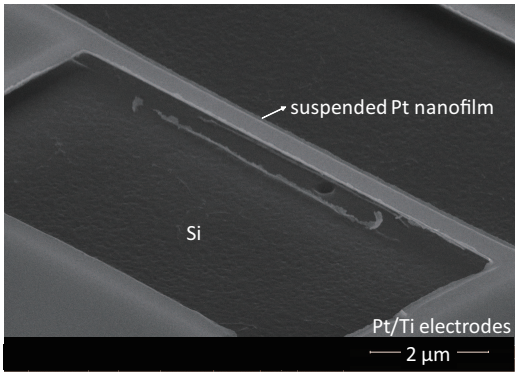


Fig. 1. SEM image of the suspended Pt nanofilm (Sample-1) at a tilted angle.

Table 1. Dimensions of the three measured suspended Pt nanofilms.

	Thickness/nm	Width/μm	Length/μm
Sample-1	39.8	0.495	9.65
Sample-2	40.0	0.622	9.44
Sample-3	40.3	0.470	9.52

3.2. Measurement system and processes

The 3ω signals were collected using a commercial lock-in amplifier (Stanford Research SR830) with a maximum output 1ω frequency of 34 kHz. The measurement system was established based on a previous study[20] and the electrical circuit is shown in Fig. 2, where the test suspended nanofilm is connected in series with an AC resistance box with 0.01 Ω resolution. The built-in signal generator of the lock-in amplifier was used to supply an AC voltage for the circuit. The electrical potentials at either end of the test sample and the resistance box were respectively differentiated via two differential amplifiers AMP-03 and then input into the A and B channels of the lock-in amplifier. All voltage signals were collected by the standard four wire method. The test nanofilms were placed in a temperature-controlled vacuum chamber (Oxford Instruments Optistat CF-V-KT) and the temperature was varied from 80–380 K at a pressure of approximately 5×10^{-4} Pa, so that the heat loss from the nanofilm to the environment could be neglected.

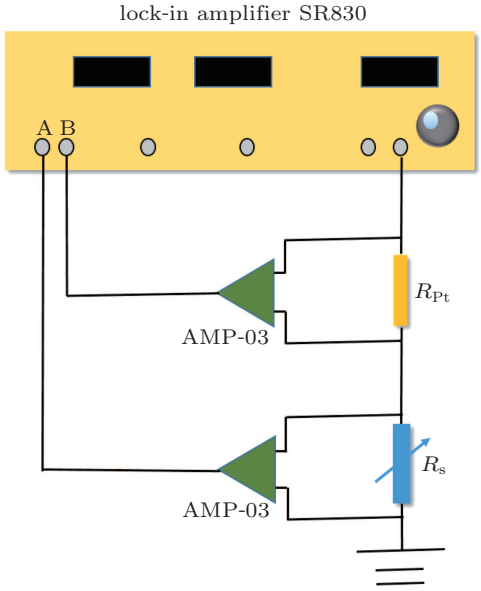


Fig. 2. (color online) Schematic of the measurement circuit.

During the measurement process, the resistance box was adjusted to equal the electrical resistance of the test Pt nanofilm. The 1ω signals of the test nanofilm and the resistance box were set to be the same, and the signal noise in the circuit was eliminated by subtracting the signals of the test nanofilm and the resistance box.[20,21] Additionally, the current was controlled to within 200 μA and the temperature fluctuation to within 5 K, so that the resistance fluctuation was less than 0.3% of the total resistance in the circuit, thus ensuring that the current source assumption was satisfied.

Before taking measurements, a suspended Pt wire of 30 μm diameter and 20.2 mm length was measured to validate the 3ω measurement system. Figure 3 shows the 3ω voltages changing with the frequency. The $V_{3\omega}$–f fitting results show that the thermal conductivity and the specific heat

were 71.6 W/(m·K) and 139 J/(kg·K), respectively, at room temperature, which are very close to the reference values of 71.4 W/(m·K) and 133 J/(kg·K).[18–21]

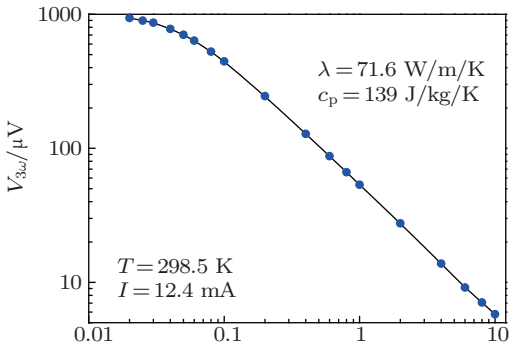


Fig. 3. (color online) The 3ω voltages changing with the input current frequency of a suspended Pt wire with 30 μm diameter and 20.2 mm length.

4. Results and discussion

4.1. Electrical resistance and thermal conductivity

According to Eq. (1), the temperature-dependent electrical resistance and temperature–resistance coefficient are prerequisites for determining the thermophysical properties of suspended nanofilms. Figure 4 shows the temperature-dependent electrical resistance of the three nanofilms, indicating a linear relationship between the electrical resistance and temperature at 80–380 K. The electrical resistance of the three samples was 191.8–265.9 Ω, within the range of previous measurements.[1–4,7–9] Because the three samples were fabricated in different PVD runs, the differences between the three samples were mainly due to variations in grain sizes in the polycrystalline nanofilms. In previous work, these were measured by atomic force microscopy (AFM) and x-ray diffraction (XRD) to be 20–40 nm for the same fabrication processes.[8,9]

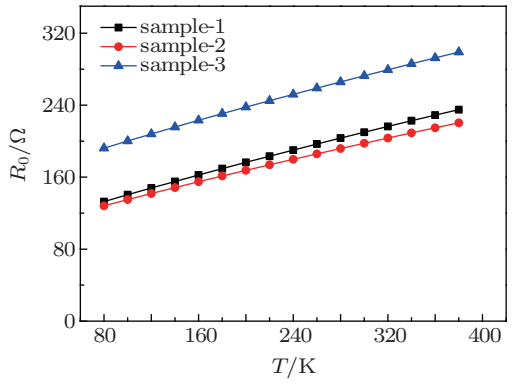


Fig. 4. (color online) Temperature-dependent electrical resistance of the suspended 40-nm-thick Pt nanofilms.

The thermal conductivities of the three samples obtained by the $V_{3\omega}$–f curve fitting method are shown in Fig. 5. The thermal conductivities of the suspended 40-nm-thick Pt nanofilms increased from ∼ 20–50 W/(m·K) as the temperature increased from 80–380 K, which is in accordance with our previous results.[1–4,7–9] The error bars indicate 5% uncertainty, which will be discussed in detail in the "Accuracy analysis" section.

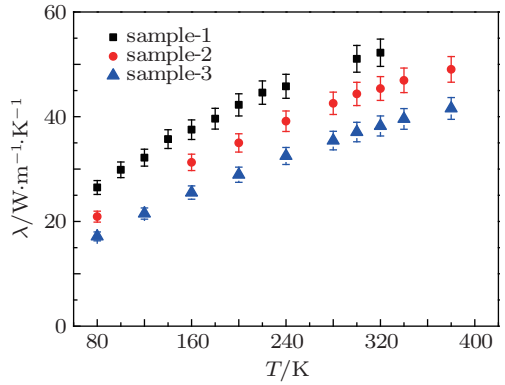


Fig. 5. (color online) Temperature-dependent thermal conductivities of the suspended 40-nm-thick Pt nanofilms.

4.2. Specific heat

Figure 6 shows the temperature-dependent specific heats of the suspended 40-nm-thick Pt nanofilms in a vacuum (∼ 5×10^{-4} Pa) and the corresponding bulk values.[22] When the temperature increases from 80–380 K, the specific heats increased to a maximum value at about 300 K and then slightly decreased, ranging from 166–304 J/(kg·K) with 10% uncertainties, much higher than the corresponding bulk values. At 300 K, the specific heats of the three samples were 301 J/(kg·K), 294 J/(kg·K), and 245 J/(kg·K), some 1.8–2.3 times the bulk value of 133 J/(kg·K). This suggests significant size effects. The ratio of the nanofilm specific heat to the bulk value, $c_{p,nanofilm}/c_{p,bulk}$, ranges from 1.65–2.60 over 80–380 K, as shown in Fig. 7. Figure 7 also shows the measured specific heats of supported Cu, Al, and Au nanofilms with thicknesses of 20–60 nm, as reported by Yu *et al.*,[12–14] Lugo *et al.*,[15,16] and Queen *et al.*[17] Although Queen *et al.*[17] reported no size effects for the specific heat of a supported 54.6-nm-thick Au nanofilm, both Yu *et al.*[12–14] and Lugo *et al.*[15,16] reported significant size effects for the specific heats of Cu, Al, and Au nanofilms. For thicknesses of 20–60 nm, $c_{p,nanofilm}/c_{p,bulk}$ ranging from 1.16 to 1.59 for supported Cu nanofilms,[12,14,16] from 1.34–1.60 for supported Al nanofilms,[12,13,15] and from 1.23–1.78 for supported Au nanofilms.[15] The present $c_{p,nanofilm}/c_{p,bulk}$ data for the suspended 40-nm-thick Pt nanofilms are comparable with the reference data reported by Yu *et al.* and Lugo *et al.*, demonstrating significant enhancement of the specific heats of PVD metallic nanofilms with thicknesses of several tens of nanometers.

According to Yu *et al.*,[10–12] the enhancement of the specific heats of metallic nanofilms can be mainly attributed to two factors. First, the softening of phonons at the surfaces or grain boundaries enhances the specific heat of a constant volume c_v, which means that the atoms at the surface or grain

boundaries need more heat input to increase the temperature. Second, the grain boundaries and defects in the nanoscale crystalline structure significantly increase the thermal expansion coefficient, causing further enhancement of c_p because the nanofilm needs more energy to perform the work of expansion. Therefore, compared with the bulk material, nanofilms need more heat input to increase internal energy and perform the necessary expansion work because of the important role of the atoms at the surfaces and grain boundaries. For the suspended nanofilms measured in the present work, the effects of the surface atoms are more significant, because there are two free surfaces, and thus the specific heats of the suspended nanofilms are even larger than those of supported nanofilms.

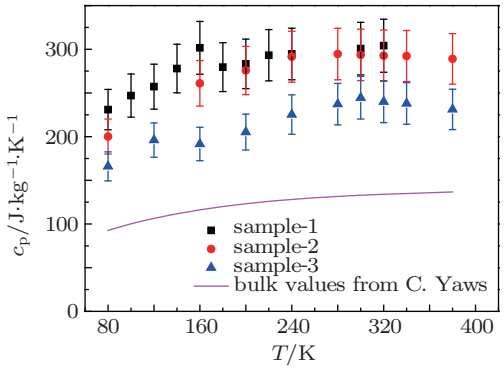


Fig. 6. (color online) Temperature-dependent specific heat of the suspended 40-nm-thick Pt nanofilms.

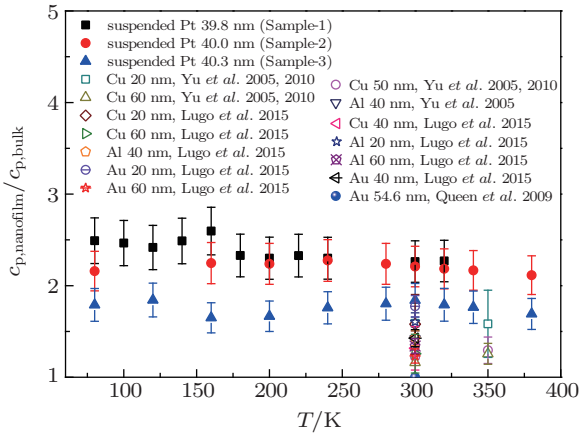


Fig. 7. (color online) Ratio of the specific heat of metallic nanofilms to the bulk value. The solid squares, circles, and triangles denote the results of the suspended Pt nanofilms measured in this work; the hollow symbols denote reference data for supported Cu, Al, and Au nanofilms (20–60-nm thick) with significant size effects ($c_{p,nanofilm}/c_{p,bulk} > 1$); spheres denote reference data for a supported Au nanofilm (54.6-nm thick) with no size effects ($c_{p,nanofilm}/c_{p,bulk} = 1$).

4.3. Accuracy analysis

The uncertainties of the dimension measurements were less than 1%. The collected 3ω signals had a 4-digit resolution and uncertainties of less than 0.3%. The main uncertainty comes from the two-parameter fitting process using Eqs. (1)–(3). Actually, the low-frequency limit of Eqs. (1)–(3) gives a constant 3ω voltage that depends only on the thermal conductivity, and the high-frequency limit gives a $V_{3\omega}$–f curve that is only dependent on the specific heat.[18] Therefore, if the 3ω voltages at full frequencies can be collected, the thermal conductivity and the specific heat can be independently extracted with high accuracy. For the nanofilm dimensions, the $V_{3\omega}$–f curve is independent of the thermal conductivity for input frequencies of $\sim$ 1 MHz. However, with the lock-in SR380 amplifier, we could only collect 3ω signals with input frequencies of less than 34 kHz. Therefore, at low frequencies, we could obtain a constant 3ω voltage that is very sensitive to the thermal conductivity, but could not obtain a $V_{3\omega}$–f curve at high frequencies that is very sensitive to the specific heat. Thus, the only way to extract the specific heat is the two-parameter fitting method, which includes a degree of uncertainty. Figures 8 and 9 show the $V_{3\omega}$–f fitting curves at various thermal conductivities and specific heats for Sample-2 at 80 K. As

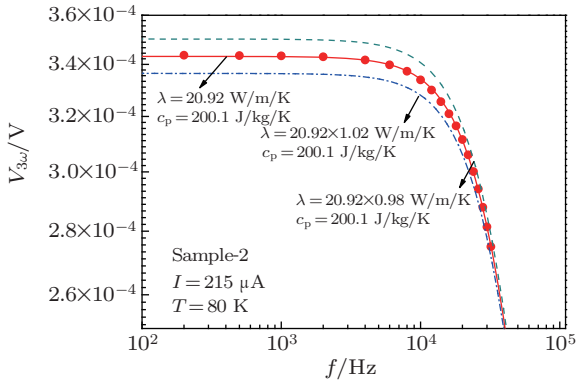


Fig. 8. (color online) $V_{3\omega}$–f fitting curves at various thermal conductivities for Sample-2 at 80 K. The solid circles show the experimental data; the solid line shows the best fit with 0.01 W/(m·K) and 0.01 J/(kg·K) resolutions, with the fitting results being 20.92 W/(m·K) and 200.1 J/(kg·K); the dashed line shows the $V_{3\omega}$–f curve with a thermal conductivity 2% lower than the fitting result, whereas the dash-dot line shows the $V_{3\omega}$–f curve with a thermal conductivity 2% higher than the fitting result.

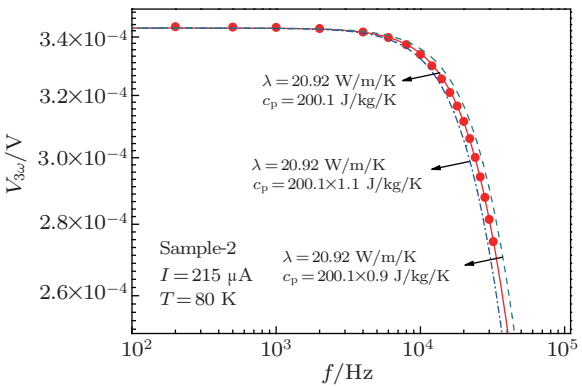


Fig. 9. (color online) $V_{3\omega}$–f fitting curves at various specific heats for Sample-2 at 80 K. The solid circles show the experimental data; the solid line shows the best fit with 0.01 W/(m·K) and 0.01 J/(kg·K) resolutions, with the fitting results being 20.92 W/(m·K) and 200.1 J/(kg·K); the dashed line shows the $V_{3\omega}$–f curve with a specific heat 10% lower than the fitting result, whereas the dash-dot line shows the $V_{3\omega}$–f curve with a specific heat 10% higher than the fitting result.

seen from Figs. 8 and 9, the uncertainty of the thermal conductivity using this fitting method is evidently less than 2%, whereas the specific heat is accurate to within 10%. Therefore, the total uncertainty of the thermal conductivity is estimated to be 5%, whereas the total uncertainty of the specific heat is 10%, as indicated by the error bars in Figs. 5–7.

5. Conclusions

This study investigated the specific heats of suspended 40-nm-thick Pt nanofilms at 80–380 K and $\sim 5\times 10^{-4}$ Pa. The specific heats of three measured samples ranged from 166–304 J/(kg·K) over 80–380 K, which are 1.65–2.60 times the bulk values. At 300 K, the specific heats of the three samples were 301 J/(kg·K), 294 J/(kg·K), and 245 J/(kg·K), some 1.8–2.3 times the bulk value, indicating significant size effects. These results are useful for scientific research in nanoscale thermophysics and the design of nanoscale devices for transient thermal response.

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