Thermal conductivity of free-standing silicon nanowire using Raman spectroscopy

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Abstract
Low dimensional systems, nanowires (NWs), in particular, have exhibited excellent optical and electronic properties. Understanding the thermal properties in semiconductor NWs is very important for their applications in electronic devices. In the present study, the thermal conductivity of a freestanding silicon NW is estimated by employing Raman spectroscopy. The advantage of this technique is that the excitation source (laser) acts as both the heater and probe. The variations of the first-order Raman peak position of the freestanding silicon NW with respect to temperature and laser power are recorded. From the analysis of effective laser power absorbed by exposed silicon NW and a detailed Raman study along with the concept of longitudinal heat distribution in silicon NW, the thermal conductivity of the freestanding silicon NW of \~{}112 nm diameter is estimated to be \~{}53 W m\textsuperscript{-1} K\textsuperscript{-1}.

Keywords: nanowire, thermal conductivity, phonon, Raman spectroscopy, thermoelectric, silicon

(Some figures may appear in colour only in the online journal)

1. Introduction
Silicon-based semiconductor technology has brought a technological revolution towards the progress of our society, which has benefited tremendously from its versatile use and remarkable capabilities. The development of electronics based on silicon and its supporting technology has brought down the physical size of devices and interconnects to the nanoscale regime. In this connection, nanowires (NWs) in general and silicon NWs, in particular, are promising materials for the miniaturization of devices towards the nanoscale regime [1, 2]. Nowadays, the growth of NWs by different methods has been achieved with great control over their crystal quality, aspect ratio and heterostructures that has opened a new window to engineer various functional properties [3–7]. In a world of ever-shrinking circuit sizes due to the integration of various miniaturized components such as NWs, the management, and transport of heat is paramount. It is quite important for better understanding and designing heat transporting devices in a host of applications, from heat management in high-power electronics, batteries, and photovoltaics to thermoelectric energy harvesting and solid-state cooling. For example, applications such as thermoelectric energy harvesting, solid-state cooling, and thermal barrier coatings would
require extremely low thermal conductivity. On the contrary, heat management in high-power electronics, batteries, and photovoltaics would require high thermal conductivity. Estimating the thermal conductivity of nanosystems will allow efficient heat management for a wide range of technological applications. The fundamental study of the thermal transport properties of silicon NWs plays a pivotal role in heat dissipation for potential heat drain applications, and future thermoelectric materials. NWs having higher electrical conductivity and lower thermal conductivity are desired for effective thermoelectric applications [8–10]. A better understanding of thermal transport in nanostructures also has direct consequences in fabricating NW-based thermoelectric devices. Moreover, in the Si NWs, the confined phonon can significantly affect the thermal conduction [11]. Thus, it is quite clear that prior determination of the thermal conductivity of the NWs is of extreme importance.

Thermal conductivity in solids is due to the heat transfer by the electrons and phonons [12]. The predominant carriers of heat in the case of semiconductors and insulators are the phonons. Reduction of the dimension of the bulk material into nanostructure results in the spatial confinement of the thermal carriers. Movement of these carriers in a confined geometry will result in increased phonon boundary scattering and phonon–phonon scattering rate. Considering these facts, thermal conductivity as a function of silicon NW diameter has been investigated using molecular dynamics simulations by Ponomareva et al. [13]. Their finding shows an interesting trend in thermal conductivity; predominant boundary scattering leads to suppressed thermal conductivity for NWs having diameter >1.5 nm and phonon confinement effect results in enhanced thermal conductivity in silicon NWs having a diameter <1.5 nm [13].

Measurement of the thermal conductivity of nanostructures such as graphene, carbon nanotubes, NWs, TMDCs is very challenging. In literature, thermal conductivity of nanostructures is reported using micro heaters [14], 3ω method [15–17], Raman spectroscopy [18–23], time-domain thermoreflectance [24]. Among these techniques, Raman spectroscopy is found to be a simple and convenient technique to measure thermal conductivity that has recently been applied to 2-D [18–21], as well as 1-D nanostructures [22,23,25]. The growing use of Raman thermometry arises from advantages lacking in other established techniques such as 3ω [15,16], micro heating [14] that are relatively complex, require direct contact with sample with the necessity of microfabrication. Unlike the other standard optical techniques that measure the thermal conductivity only in the cross-plane direction, Raman technique measures the in-plane thermal conductivity. As it has been found in literature that for nanostructures having anisotropy in particular directions, the measurement of in-plane thermal conductivity is more relevant [26]. In Raman scattering method, the laser acts both as heating and excitation source [27–29]. Analyzing the shift of the Raman peak position caused by uniform rise in temperature and laser power induced local heating, the thermal conductivity can be estimated.

In the literature, different methods of measurement of the thermal conductivity of silicon NW such as Joule heating [14] or Raman thermometry method [23] have been reported. The Joule heating method needs complex experimental procedure (test sample fabrication) and also the interfacial contact resistance could be a significant source of error while determining the thermal conductivity. In the Raman thermometry method, thermal conductivity measurement on the cantilevered silicon NW by Doerk et al considers linear regression of radially averaged temperature difference along the axis of NW [23].

In this work, we have used a new and relatively simple method to measure the thermal conductivity of the freestanding isolated silicon NW by using Raman spectroscopy which includes the temperature-dependent as well as the power-dependent Raman spectra. The first-order temperature coefficient \(\delta \omega/\delta T\) is obtained from the temperature-dependent Raman study and variation of Raman peak with laser power \(\delta \omega/\delta P\) is determined by power-dependent Raman study and further analysis is carried out to calculate thermal conductivity. It should be mentioned that this adopted method for the thermal conductivity measurement was successfully able to measure the thermal conductivity of the two-dimensional systems, e.g. graphene, few layers of MoS\(_2\), etc., as per the previous reports [18,19]. In this report, we have extended this method for the measurement of thermal conductivity in one dimensional system.

2. Experimental

Silicon wafer containing silicon NWs is commercially procured from CVD Materials Corporation, USA that has grown by VLS CVD using First Nano\textsuperscript{TM} Easytube\textsuperscript{®} system. Microstructure characterization of silicon NWs is performed using Field Emission Scanning Electron Microscope (Zeiss make). Raman spectra silicon NWs are recorded using a confocal micro-Raman spectrometer (LABRAM HR Evolution, Horiba-Jobin-Yvon, France) with the 532 nm excitation of frequency-doubled Nd:YAG laser in the backscattering mode with an 1800 grooves/mm grating. The temperature-dependent Raman studies are carried out using a temperature stage (LINKAM THMS 600, Linkam Scientific Instruments, UK) with the help of a long working distance 50X objective. The power-dependent spectra in ambient temperature conditions are collected with a 100X objective (NA = 0.9) by engaging discrete neutral density (ND) filters. For each ND filter, the final power is measured at the microscope objective end with a power meter (Thorlabs, USA). The surface roughness of the silicon NW is checked using atomic force microscopy (AFM) and high-resolution transmission electron microscopy (TEM). AFM studies are carried out using Digital Instruments NanoScope III with Budget Sensors’ Tap300-G tip in the tapping mode. The tips are uncoated and have a resonant frequency of 300 kHz. HRTEM images of silicon NW is taken with JEOL 300 with operating voltage 300 kV.

Dense silicon NWs growth with a diameter varying between 100 and 500 nm with a large length (of several microns) can be easily noticed from figure 1(a). Raman spectra are recorded on a bunch of NWs and a strong first-order
Raman peak of silicon is observed as shown in figure 1(b). It may be noted that the width of the Raman spectra is wider than bulk silicon. These NWs from the substrate are then transferred to a carbon-coated copper TEM grid. Once transferred, the grid is inspected for isolated single freestanding NW. This isolated individual NW can sometimes found to be suspended on ruptured carbon film where one end rests on the carbon film and another end lies on the copper grid. To minimize the role of contact on thermal conductivity we consider a suspended single NW of length much larger than the diameter of the laser spot. Figure 3(a) shows an SEM image of such a freestanding silicon NW of diameter ~112 nm with a suspended length of ~7 µm. A careful analysis of the freestanding isolated NW indicates that the diameter of the NW is not uniform throughout its length, i.e. from one side (right side) its diameter gradually decreases. Such tapering of diameter is expected for CVD grown large length NWs. However, for the present study for thermal conductivity measurement, the diameter can be approximated to be uniform. The suspended NW, laser heating are schematically shown on the left side of figure 3(b). We also schematically present the effective cross-sectional area of NW exposed to laser on the right side of figure 3(b). It can be clearly seen that the diameter of the laser spot is much larger as compared to the diameter of the suspended NW (details of which will be explained in a later section). To minimize the experimental uncertainty in Raman measurement, the high-temperature studies are deliberately avoided in the present case because at higher temperatures silicon nanowire (NW) samples could undergo oxidation resulting in a core–shell structure of Si/SiO₂. This can affect the value of first-order temperature coefficient (α) of the silicon Raman mode. Furthermore, all the Raman measurements are done at the lowest possible laser power to avoid local heating. This is achieved by monitoring the silicon Raman mode at ~521 cm⁻¹ in the real-time display mode and then varying the laser power. In all the temperature dependent Raman measurements, before taking the measurement at any specific temperature, sufficient time is given in order to equilibrate the stage temperature, thus reducing temperature uncertainty. Also, the cooling/heating ramp rate is kept very low. The Raman spectra are collected from 77 K to 298 K. The acquired spectra are fitted with Lorentzian lineshape to obtain the Raman peak positions and peak width (FWHM).

3. Results and discussion

Thermal conductivity measurement using Raman spectroscopy would result in an inaccurate estimation for a sample directly placed on a substrate. This is due to the fact that as a part of the heat will dissipate to the substrate. In view of this most of the measurements have been carried out on a free-standing sample [18,19,22]. As reported thermal conductivity estimation by Raman scattering requires a temperature and laser power-dependent studies. First, we carried out a temperature-dependent Raman study. The temperature of the sample is varied from 77 to 298 K. Figure 4(a) shows the temperature-dependent Raman spectra. A systematic redshift of the first-order Raman peak position is noticed for the freestanding silicon NW with increasing temperature. This redshift is due to both the volume and temperature contribution as a consequence of anharmonicity of lattice vibration. The Raman peaks are well fitted with the Lorentzian lineshape and the extracted Raman peak position with the temperature is plotted in figure 4(b). The peak position as a function of temperature shows a linear relationship as fitted using ω = ω₀ + α · T, where ω₀ is the frequency at T = 0 K, α (slope of the curve) is the first-order temperature coefficient [18,22]. The value of α is found to be −0.01776 cm⁻¹ K⁻¹. Doerk et al [30] have studied the variation of first-order temperature coefficient as a function of the diameter of silicon NW ranging from 20 to 200 nm. The diameter dependent first-order temperature coefficient does not change significantly and our observed first-order temperature coefficient is also consistent with their reported result [30].

The broadening of the linewidth which is the measurer of phonon lifetime can be explained using the decay of phonon into two phonons or three phonons processes satisfying the conservation law [31,32]. Considering a three phonons process, the line width can be expressed as,

$$\Gamma(T) = \Gamma_0 + E \left[ 1 + \frac{2}{e^{\omega_0} - 1} + F \left[ 1 + \frac{3}{e^{\omega_2} - 1} + \frac{3}{(e^{\omega_2} - 1)^2} \right] \right]^2,$$

where $x = \frac{\hbar \omega}{2 k_B T}$, $y = \frac{\hbar \omega}{3 k_B T}$, $\Gamma_0$ is FWHM of the silicon NW in low temperature and $E$, $F$ is cubic and quartic anharmonic constants respectively [33]. The variation of FWHM as a function of temperature is well fitted by the above equation for three phonon decay (figure 4(b)) with cubic and quartic anharmonic constants of −0.3097 and 0.4375, respectively.

Laser power dependent Raman studies with the variation of power is analogous to the temperature-dependent Raman spectra, it signifies that a strong laser power on the sample results in the localized heating [19]. Here we want to point out that a careful determination of the effective diameter of the laser beam spot ($D_l$) and actual power absorbed by the NW are desired for the precise thermal conductivity measurement. A theoretical estimation of the diffraction-limited spot size ($D_l = 2\lambda/\pi a_{NA}$) puts the lower limit on the spot size to be 0.38 µm where $\lambda$ is the wavelength of the incident light (532 nm), NA is the numerical aperture of the objective lens (0.9), respectively. In the present study, we consider the spot size to be ~0.5 µm which is much larger than the NW diameter (112 nm). It is evident that the dimension of the laser beam spot is too large compared to the diameter of silicon NW and hence the power absorbed by the silicon NW is not the same as the incident laser power. During the experiment, the NW was placed carefully under the laser spot such that the diameter of the laser ($D_l$) spot coincides with the NW, as schematically shown in figure 3(b). Under such geometry, the cross-sectional area of the silicon NW illuminated by the laser is $D_l \cdot D_{nw}$, where $D_{nw}$ is the diameter of the silicon NW. As mentioned our laser spot size is much larger than the diameter of silicon NW, we followed the effective power calculation of Doerk et al [23]. The power absorbed ($P_{abs}$) by the silicon NW
Figure 1. (a) SEM image of silicon NWs and (b) corresponding room temperature Raman spectra.

Figure 2. (a) AFM image of multiple silicon NWs. (b) Three dimensional view of AFM image of single silicon NW with the inset showing the color plot of NW. (c) TEM image of silicon NW with inset showing the 3-D surface plot of the region bounded by yellow lines. (d) Room temperature Raman spectra of Bulk Si and single silicon NW.

\[ P_a = \frac{4 \cdot D_{nw} \cdot D_l}{\pi \cdot D_l^2} \cdot \frac{\pi}{A \cdot P_l}. \]  

It may be noted here that the actual laser power as measured under the objective lens is varied from 43 \( \mu \)W to 1.2 mW during the experiment. The absorbance \( A \) can be deduced using the formula:

\[ A = 1 - \left( \frac{\pi}{n+1} \right)^2 + \frac{\pi}{n+1} \cdot k, \]

where \( n \) and \( k \) are the refractive index and extinction coefficient, respectively [34]. From the literature, the values of \( n \) and \( k \) are found to be about 4.15 and 0.052 for bulk silicon [35] corresponding to 532 nm radiation. Considering these values the absorbance \( A \) of the silicon NW is estimated to be \( \sim 62\% \). Using the above formula, the effective power \( P_a \) is calculated to be in the range of 7 to 201 \( \mu \)W. Figure 5(a) shows the Raman spectra of the freestanding NW as a function of effective laser power.

The symmetrical broadening of the Raman line and shifting of the first-order Raman peak position towards the lower wavenumber side is also observed. The variation of Raman peak position with different power is shown in figure 5(b) and is
fitted by a straight line and the slope \( \frac{\delta \omega}{\delta P_a} \) of \( \omega \sim P_a \) is found to be \(-0.05721 \, \text{cm}^{-1} \, (\mu \text{W})^{-1} \) from the linear fit.

Here, we would like to emphasize that silicon is always associated with a native oxide layer irrespective of its synthesis methods. The thickness of the oxide layer can vary depending on process conditions. The role of the oxide layer is very important in determining various properties of silicon NW. For an instant, during the electrical transport measurement of isolated silicon NW, the native oxide layer is removed carefully before making the electrical contacts. Being an insulator and highly dielectric material, the presence of silicon dioxide (SiO\(_2\)) layer on silicon NW can significantly affect its thermal properties. It has been experimentally observed that one requires higher laser power to get a significant Raman shift in silicon NW having a thicker oxide layer analogous to the thin oxide layer covered silicon NW. The work by Piscanec et al. [36], Zixue Su et al. [33] shows shifts in the Raman spectra for both thin (~2 nm) and thick (~10 nm) oxide layers covering silicon NW respectively. In our case, significant changes in power-dependent Raman spectra are indicative of the existence of a very thin (~2–3 nm) oxide layer covering the silicon NW.

Thermal conductivity of a sample can be determined by considering the heat distribution, i.e. (i) radially heat distribution from the middle position of the sample towards the heatsink if the diameter of the sample is larger than the diameter of the laser spot (ii) longitudinal heat distribution from the middle position of the sample towards the heatsink if the diameter of the sample is comparable to the diameter of the laser spot [18]. Transport of heat through the surface of a sample with a cross-sectional area \( S \) is formulated as,

\[
\frac{\partial Q}{\partial t} = -\kappa \cdot \oint \nabla T \cdot d\Sigma, \tag{3}
\]
where $\kappa$ is the thermal conductivity of the sample, $\frac{\partial Q}{\partial t}$ is the rate of heat transferred through the sample, $\nabla T$ is the temperature gradient. When the diameter of the sample is comparable to the diameter of the laser spot, the thermal conductivity of the sample can be formulated from the equation (3) as $\kappa = \frac{L}{\pi S \Delta P \Delta T}$, where $L$ is the distance from the laser-heated position of the sample to the heat sink and $\Delta T$ is the change in temperature due to the variation of the laser power [18]. The value of $L$ is about ~3.4 $\mu$m as measured from the SEM image [figure 3(a)]. We note that in our case the diameter of the laser spot is quite larger than the diameter of the silicon NW. However, if we consider the portion of the laser power ($P_a$) absorbed by the silicon NW as shown in figure 3(b), then the heat distribution will be longitudinally allowing us to use the formula: $\kappa = \frac{L}{\pi S \Delta P \Delta T}$. We assumed that the increase in the laser power, i.e. increasing the local temperature of the silicon NW does not affect the temperature of the heat sink and the local heat generated is transferred uniformly to the heat sink. With the help of linear fit of the $\omega \sim T$ graph, i.e. $\omega = \omega_0 + \alpha \cdot T$, the above formula can be modified to $\kappa = \frac{L}{\pi S \Delta P} \cdot \alpha \cdot \left( \frac{\delta \omega}{\delta P_a} \right)^{-1}$.

With laser spot diameter 0.5 $\mu$m, the diameter of silicon NW 112 nm, length of silicon NW from the laser spot to heat sink ~3.4 $\mu$m, the value of first-order temperature coefficient ($\alpha$) as $-0.01776 \text{ cm}^{-1} \text{ K}^{-1}$ and value of $\frac{\delta \omega}{\delta P_a}$ as $-0.05721 \text{ cm}^{-1} (\mu \text{W})^{-1}$, the thermal conductivity of the single silicon NW sample is estimated to be ~53 W m$^{-1}$ K$^{-1}$. In this calculation, we used the absorbance ($A$) value of bulk silicon which is ~62% as there exists no experimental reports on absorbance value of silicon NW.

Doerk et al reported the variation of thermal conductivity with NW diameter and for the silicon NW with diameter ~115 nm, room temperature thermal conductivity is about 40 W m$^{-1}$ K$^{-1}$ [23]. Similar thermal conductivity values were also reported by Li et al for individual NWs with a nearly similar diameter [14]. Although our thermal conductivity estimation is in broad agreement with that of Doerk et al.
[23] and Li et al [14], our estimated value is slightly higher. We now discuss the various plausible reasons for this small discrepancy in thermal conductivity value. Thermal conductivity of a material is highly dependent on the external strain which affects the phonon group velocity and thermal properties. Equilibrium Molecular Dynamics calculations by Li et al on thermal conductivity of silicon NWs as a function of strain reveal higher thermal conductivity under compressive strain and lower thermal conductivity under tensile strain as compared to the unstrained one [37–39]. It is well known that Raman spectroscopy is a non-destructive method for measuring the tensile and compressive strain present in the sample. Tensile strain and compressive strain in the material affect the phonon mean free path which results in Raman peak shift as compared to the pristine one. To examine the effect of strain in our case, Raman spectra on bulk Si and silicon NW are recorded under the same conditions. To exclude the localized heating induced Raman peak shift, the lowest laser power is chosen to figure out the effect of strain on Raman spectra. Raman spectra of bulk Si and silicon NW are depicted in figure 2(d). The unaltered Raman peak position in both bulk Si and silicon NW confirms the absence of strain in the NW. Another factor that can greatly influence the thermal conductivity of the silicon NW is surface roughness/corrugation [40,41]. In order to measure the average surface roughness present in the NW, we characterized the NWs using both HRTEM and AFM. The AFM characterized image of silicon NW is depicted in figure 2(a) and (b). From the AFM images, the average roughness of the silicon NW is found to be ~2.4 nm. Figure 2(c) shows the TEM image and the selected region is analyzed by ImageJ software for further roughness calculation. Considering the baseline in the surface plot of the TEM image as shown in the inset of figure 2(c), the average roughness is about ~3 nm, which is consistent with the measured roughness from AFM. When compared to the diameter of the silicon NW, this measured roughness is quite negligible. Therefore, we rule out any significant effects of roughness on the thermal conductivity values. We emphasize here that the NWs used by Poborochi et al are quite thin (~55 nm) and hence the role of small corrugations is quite dramatic in reducing the NW thermal conductivity[41]. In our case, as discussed already, the NWs are much bigger in diameter (~112 nm) and the roughness is of the order of ~3 nm only. Therefore, we conclude that both strain and surface roughness do not play a significant role in the small discrepancy of increased thermal conductivity in our case. In order to reconcile with the slightly higher value of thermal conductivity, we may recall that one of the factor that appears in our working formula is the laser heating induced Raman peak shift, the thermal conductivity of the free-standing single silicon NW is found to be ~53 W m⁻¹ K⁻¹ by using the bulk silicon absorbance values. By using previously calculated absorbance value, the thermal conductivity value thus obtained reduces to ~43 W m⁻¹ K⁻¹. Exact experimental determination of the absorbance of individual silicon NW would help in reducing this uncertainty.

4. Conclusions

In summary, Raman spectroscopy is employed to determine the thermal conductivity of the CVD grown free-standing silicon NW of diameter 112 nm. Free-standing silicon NWs are obtained by transferring the NWs from the substrate to a carbon-coated copper TEM grid. Isolated individual NW can sometimes found to be suspended on ruptured carbon film where one end rests on the carbon film and another end lies on the copper grid. To minimize the role of contact on thermal conductivity we consider a suspended single NW of length much larger than the diameter of the laser spot. The temperature dependence of Raman spectra provides the first-order temperature coefficient (α) which is ~−0.01776 cm⁻¹ K⁻¹ and laser-induced Raman spectra undergo a redshift which provides a slope of ~−0.05721 cm⁻¹ (μW)⁻¹. By means of the first-order temperature coefficient (α) value and slope of the Raman peak position − power graph, the thermal conductivity of the free-standing single silicon NW is found to be ~43 W m⁻¹ K⁻¹. We also thank two anonymous reviewers for their feedback in improving the quality of the manuscript.

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