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Multiscale Phonon Thermal Transport in Nano-Porous Silicon

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We performed a comprehensive *multi-scale* phonon-mediated thermal transport study of nano-porous silicon (np-Si) films with average porosities in the range of $\phi = 30 - 70\%$. This depth-resolved thermal characterization involves a combination of optical methods including femtosecond laser-based time-domain thermo-reflectance (TDTR) with MHz modulation rates, opto-thermal micro-Raman spectroscopy and continuum laser wave-based frequency domain thermo-reflectance (FDTR) with kHz modulation rates probing depths of studied samples over 0.5 - 1.2, 2 - 3.2 and 23 - 34 μm , respectively. We revealed a systematic decrease in thermal conductivity (k) with the rise of ϕ , i.e. with the lowering of the Si crystalline phase volumetric fraction. These data were used to validate our semi-classical phonon Monte-Carlo and finite element mesh (FEM) simulations of heat conduction, taking into account disordered geometry configurations with various ϕ and pore size, and laser-induced temperature distributions, respectively. At high ϕ , the decrease in k is additionally influenced by the disordering of the crystal structure, as evidenced by the near-surface sensitive TDTR and Rutherford Backscattering Spectroscopy (RBS) measurements. Importantly, the k values measured by FDTR over larger depths inside np-Si were found to be anisotropic and lower than those detected by the near-surface sensitive TDTR and Raman thermal probes. This finding is supported by the cross-sectional scanning electron microscopy (SEM) image indicating enhanced ϕ distribution over these micron-scale probed depths. Our study opens an avenue for nano-to-micro-meter scale thermal depth profiling of porous semiconducting media with inhomogeneous porosity distributions applicable for efficient thermoelectric and thermal management.

Effective thermal management at nano- and micro-scales has become critical for various applications involving nano- and micro-electronics and advanced energy transport and conversion. The emergence of 3D integration of devices has underscored the significance of developing materials with low thermal conductivity k that are also compatible with CMOS technology [1]. Understanding heat transfer in nanostructured materials is important for their integration into nanoelectronics and energy conversion devices like thermoelectric generators and coolers [2, 3]. Nano-porous silicon (np-Si), fabricated through the electrochemical etching of single crystalline silicon wafers (c-Si), exhibits k that is significantly lower, approximately two-to-three orders of magnitude, compared to that of c-Si. This lower k is a result of phonon scattering across pore surfaces within complex porous architectures and increased phonon-phonon scattering due to its nanoscale architecture [4]. For this reason, np-Si holds significant technological value as a

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nanostuctured medium for thermoelectrics and numerous on-chip applications requiring thermal isolation. A noteworthy illustration can be found in the realm of Si sensors and Si-based micro-electromechanical devices [5, 6]. The remarkably low k of highly porous Si forms the foundation for various potential applications, including thermoelectric conversion [7, 8], thermal sensors [9, 10], thermally induced broadband sound emission [11], and mid-infrared applications [12]. Moreover, optimization of its use in optoelectronic applications, given its remarkable photoluminescent characteristics, requires a good knowledge of its thermal properties. Consequently, this topic has become of much interest in the realm of nanoscale heat transport [13, 14]. It is acknowledged that thermal depth profiling is very important for characterization of heat conduction in layered and inhomogeneously structured materials where adhesion, near-surface degradation, corrosion and phase transformation can take place [15 - 18]. Thus, a more detailed understanding of thermal depth profiling in np-Si can have valuable practical consequences, because the depth porosity variations impact the phonon transport properties, consequently affecting k value. In its turn such depth dependent variation of k opens broader adjustment possibilities, which can be of interest in thermoelectric and thermal management applications.

The fundamental approach to heat transport measurements consistently involves introducing a heat source that disperses within and around the sample. This process entails tracking the dispersion of heat and then comparing it to a model founded on the principles of thermal diffusion to extract the targeted thermal properties. Various thermal metrologies such as photo-acoustics [19, 20], 3ω [21, 22], lock-in thermography [23], optical pump-probe method [24], have been employed to study k as a function of porosity, the morphology and structure [25] of the np-Si samples, the type of initial c-Si substrate [26], its resistivity [27] and the electrochemical fabrication [28]. The micro-Raman spectroscopy and scanning thermal microscopy were used to extract the local k of several types of meso-porous and nanostructured Si materials [29 - 33]. Lock-in thermography on np-Si demonstrated thermal anisotropy in cross-plane versus in-plane directions due to the higher interconnection of the porous structure along the etching direction [23, 34].

However, there have not been prior studies on *multi-scale depth profiling of thermal transport in np-Si*. For that reason, in this work we performed a comprehensive *multi-scale* phonon-mediated thermal transport study of np-Si films with average porosities in the range of $\varphi = 30 - 70 \%$. This depth-resolved thermal characterization involves a combination of optical methods including femtosecond laser-based time-domain thermo-reflectance (TDTR) with MHz modulation rates, opto-thermal micro-Raman spectroscopy and continuum laser wave-based frequency domain thermoreflectance (FDTR) with kHz modulation rates probing depths of studied porous media over 0.5 - 1.2, 2 - 3.2 and 23 - 34 μm , respectively. In TDTR and FDTR approaches the heat penetration depth was controlled by modulating the laser pump beams at 1 - 10 MHz and 1 - 100 kHz rates, respectively. The micro-Raman spectral thermometry-based heat conduction measurements were used to validate FEM simulations of temperature distribution resulting from laser-induced heating in np-Si films. The semi-classical Monte-Carlo simulations of phonon transport were conducted with various disordered geometries to assess the impact of the corresponding porosities and nano-pore size distributions.

In our study, the np-Si films were prepared by subjecting p-type boron-doped (100)-oriented c-Si wafers with a resistivity of $\sim 10\text{-}20 \text{ m}\Omega\cdot\text{cm}$ to electrochemical anodization. The values of $\varphi = 30 - 70 \%$ in the fabricated np-Si film samples are assessed by gravimetric method. The

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thicknesses of films and pore sizes are measured to be in the range of 23-34 μm and $D = 13\text{-}25$ nm, respectively as determined by SEM (FIG. 1, 4, 6). The fabrication process as well as the procedure for conducting Raman spectroscopy measurements are provided in **Supplementary Materials**.

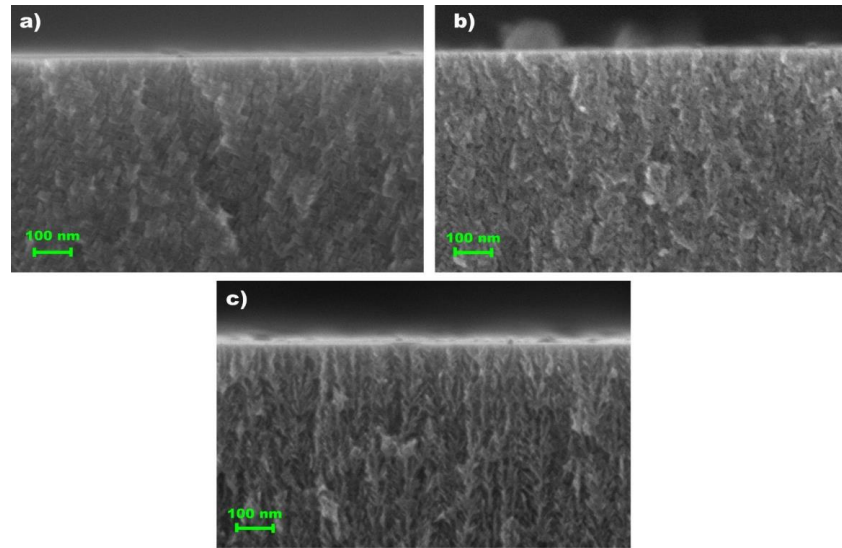


FIG.1. High resolution cross-sectional SEM images of np-Si samples with the average porosities $\varphi =$ (a) 30%, (b) 50% and (c) 70%.

The primary Raman peak at $\sim 520 \text{ cm}^{-1}$, associated with the cubic Si LO phonon mode in np-Si, as shown in FIG. 2(a), undergoes a frequency downshift with the rise of 532 nm wavelength incident optical power delivered by laser beam focused on studied samples down to a spot having 2 μm diameter. The resulting local laser-induced temperature rise was determined from measured Raman Stokes/anti-Stokes integrated peak intensity ratio [35]. FIG. 2b demonstrates the corresponding changes in temperature T induced by laser irradiation of np-Si films exhibiting φ variations. Films with higher φ experience greater heating compared to those with lower φ , attributed to the reduced volume fraction of highly conductive c-Si phase in np-Si samples. The measured local temperature values were used to validate our 3D FEM simulations of photo-induced temperature excursions in np-Si performed using the Ansys Lumerical heat module (FIG. 2c). The steady-state Fourier heat conduction equation was solved numerically using absorbed laser light through the np-Si depth (2 – 3.2 μm) for the samples with $\varphi = 30 - 70 \%$. The optical absorption depth values were extracted by assessing the absorption coefficient values obtained from both literature and our own ellipsometry data (refer to the **Supplementary Materials** for detailed explanations of the ellipsometry measurement process and assessment of the absorption depth values). The ellipsometry measurements required the knowledge of the real and imaginary parts of the refractive index which were found using Bruggeman Effective Medium Approximation (EMA) [36] which took into account the dielectric constant of air. The resulting temperature distribution, caused by laser heating in np-Si, was assessed with the k values of the

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np-Si films used as single-fitting parameters. Since the thicknesses (23 to 34 μm) of our samples are much larger than the diameter of the incident laser beam (2 μm) used in Raman thermometry, the incident laser beam was treated as a localized heating source resulting in nearly hemispherical temperature distributions across the np-Si films.

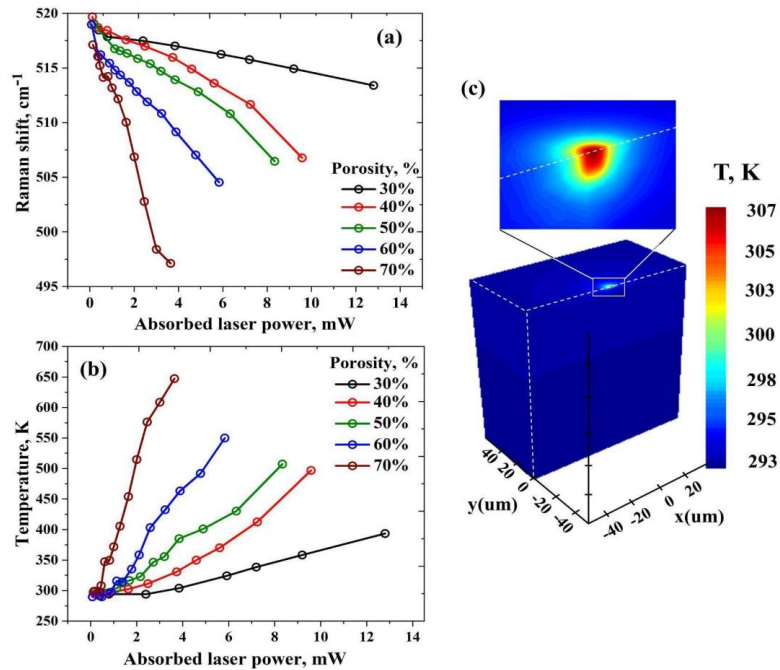


FIG.2. (a) Laser-induced Raman shifts and (b) corresponding measured local temperatures in np-Si layers with various porosities. (c) Typical FEM simulated spatial temperature distribution in np-Si with 30% porosity.

RBS/C (channeling) and RBS/R (random) measurements, which probe microstructure down to a depth of $\sim 1 \mu\text{m}$, were employed to assess how changes in crystalline structure and oxidation degree impact *near-surface* k values. In samples with low porosities ($\phi = 30 - 50 \%$), the crystalline structure remains unaltered. However, at $\phi \geq 60\%$ the disorder in the crystalline lattice becomes quite substantial, as seen from the elevated magnitude of the RBS/C spectrum for the samples with $\phi = 60 \%$ and 70% as seen in FIG.3.

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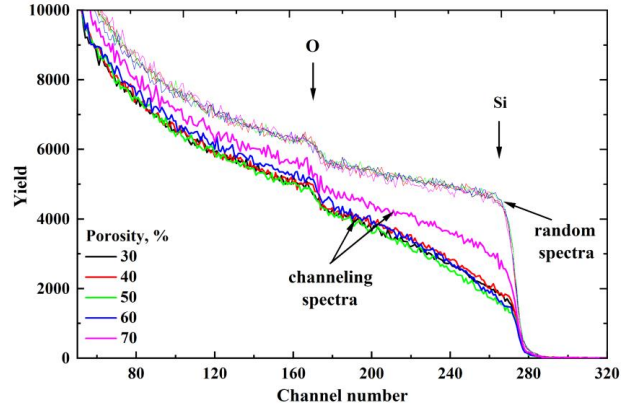


FIG.3. RBS spectra of np-Si samples having different porosities.

Subsequently, TDTR measurements, probing depths at 0.5 - 1.2 micrometers (depending on the porosity level, TABLE 1), reveal a decrease in k values with the rise of φ (excluding that for the $\varphi = 30\%$) - see FIG. 7 below. The decrease in the near-surface k in highly porous samples is connected to both the decrease of the c-Si volume fraction and the emergence of disorders in the crystalline lattice. However, at low porosities, the reduction in the volumetric fraction determines the change of the k values. Furthermore, the 70% porous sample exhibits an O/Si ratio of 0.3/0.7, whereas other porosities maintain an equal O/Si ratio of 0.25/0.75, as evident from the derived RBS/R spectra results (FIG.3) using SIMNRA code [37]. The 70% porous sample will likely be covered by the native oxide layer. However, this top layer has thickness < 2 nm and it is non-continuous on the top surface of np-Si, i.e. does not cover the entire film surface. Therefore its effect on thermal transport of underlying np-Si samples is negligible [29] since all our measurement techniques probe heat conduction on a much deeper scale, as seen in TABLE 1. The details of the TDTR, RBS/C and RBS/R methodologies are described in **Supplementary Materials**.

TABLE I. Approximate thermally probed domain volumes (heat penetration depth \times laser beam area) probed by the TDTR, FDTR, Monte Carlo and Raman-FEM techniques (computational domain areas) for various.

Methods	30%	40%	50%	60%	70%
TDTR	$(1.25 \times 10^{13}) \pi \mu\text{m}^3$	$(0.94 \times 10^{13}) \pi \mu\text{m}^3$	$(0.86 \times 10^{13}) \pi \mu\text{m}^3$	$(0.72 \times 10^{13}) \pi \mu\text{m}^3$	$(0.49 \times 10^{13}) \pi \mu\text{m}^3$
FDTR	$(27.8 \times 10^2) \pi \mu\text{m}^3$	$(34 \times 10^2) \pi \mu\text{m}^3$	$(23 \times 10^2) \pi \mu\text{m}^3$	$(30.7 \times 10^2) \pi \mu\text{m}^3$	$(31.5 \times 10^2) \pi \mu\text{m}^3$
Raman-FEM	$(2 \times 10^2) \pi \mu\text{m}^3$	$(2.3 \times 10^2) \pi \mu\text{m}^3$	$(2.4 \times 10^2) \pi \mu\text{m}^3$	$(3 \times 10^2) \pi \mu\text{m}^3$	$(3.2 \times 10^2) \pi \mu\text{m}^3$
Monte-Carlo	$0.5 \times 1 \mu\text{m}^2$	$0.5 \times 1 \mu\text{m}^2$	$0.5 \times 1 \mu\text{m}^2$	$0.5 \times 1 \mu\text{m}^2$	$0.5 \times 1 \mu\text{m}^2$

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FDTR measurements were conducted to assess the k values on the depths scale of *tens of* μm inside the np-Si films. Obtained k values are found to be lower than those probed in the near-surface ($\sim 1 \mu\text{m}$) regions as assessed by TDTR and Raman. The reduced k is associated with an enhanced distribution of porosity throughout the entire thickness of the np-Si, as illustrated in FIG. 4, where the dark vertical lines correspond to pore agglomerates. In order to see if thermal anisotropy is present in np-Si samples, we conducted sensitivity analysis presented in FIG. S6b of the **Supplementary Materials**. A good sensitivity to both cross-plane thermal conductivity k_z and anisotropy ratio $\eta = k_r/k_z = 1$ (the ratio of in-plane to cross-plane thermal conductivities) is observed. Thus, FDTR data is analyzed using three fitting parameters, i.e. k_z , η and R_0 . As a result, in FIG. 7 in addition to other thermal transport measurements and Monte-Carlo simulations, we also present our FDTR results by showing both k_r and k_z values versus porosity in np-Si samples. Almost all samples demonstrate some thermal anisotropy defined by $\eta \sim 0.6$. The difference in anisotropy values compared to the previous works [23, 34] explained by the difference in the etching fabrication process and thermal measurement methods.

In both TDTR and FDTR measurements, for accurate determination of heat conductivity, an EMA model was used (defined by Eq. S4 of the **Supplementary Materials**) to take into account the specific heat capacity of air present in the pores of np-Si samples.

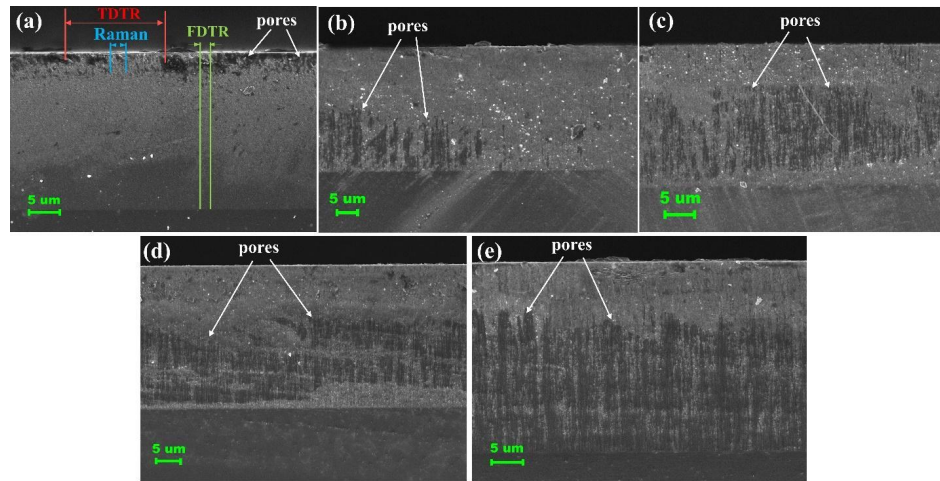


FIG. 4. Low - magnification cross-sectional SEM images of np-Si samples with $\phi =$ (a) 30%, (b) 40%, (c) 50%, (d) 60% and (e) 70%

Regarding the 30% porous sample, the significant variation in k values (FIG.7) can be attributed to the subsurface non-uniform distribution of pore agglomerates both with respect to depth and radial distribution as can be seen from FIG. 4a. TDTR measurements were conducted over small depths, but at larger lateral areas compared to other methods. Consequently, the significant low k value, measured by TDTR, can be attributed to the probed large pore volume fractions, as numerous pore agglomerates in this 30% porous sample are located near the surface.

Thermally probed domain volumes probed by the TDTR, FDTR, Monte Carlo and Raman-FEM techniques for different porosities can be found from TABLE 1.

A Monte Carlo (MC) method has been adopted for a semi-classical particle-based description of phonon transport in np-Si. The simulation domain is populated with nanostructured features as shown in FIG. 5. We use the ‘single-phonon MC approach which differs from the multi-phonon MC approach described earlier [38 - 47] and in the **Supplementary Materials** in terms of phonon attributes book-keeping. The ‘single-phonon’ method is computationally simpler. By eliminating the need to track multiple phonon positions concurrently, it reduces both the computational complexity and the required resources. [48, 49, 43 - 46].

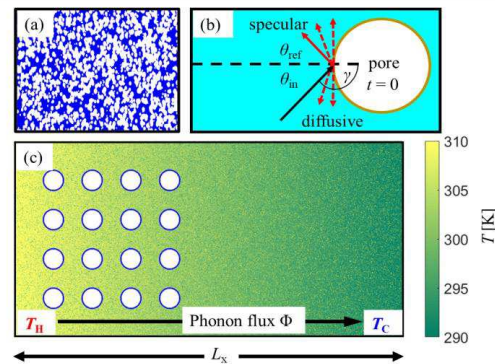


FIG.5. (a) Example of actual simulated geometry of np-Si with $\varphi = 60\%$. White regions depict porous (air) regions with no phonon transmission while blue regions depict regions of c-Si phase across which phonons can propagate. (b) Schematic of scattering mechanism for pore scattering, indicating the pore boundary, the initial angle of the phonon θ_{in} and potential new angle of propagation θ_{ref} depending on specularity parameter p . Probable paths of the phonon after scattering for both diffusive (red dashed lines) and specular (red solid line) are also depicted. (c) Schematic of the simulation domain with an example of ordered nano-porous geometry. The coloring profile indicates the established thermal gradients when the left and right contacts are set to $T_H = 310$ K (yellow) and $T_C = 290$ K (green), respectively.

In short, phonons are initialized in the contacts only, based on polarization, frequency, group velocity, and energy. Phonon probabilities are determined by the phonon dispersion relation $\omega(q)$, in accordance with the Bose-Einstein statistics at a given temperature. We use $\omega(q)$ and corresponding group velocities $v_g(q)$, as described by Pop *et al.* [41]. Phonons then propagate through the simulation domain and alternate between durations of free flight and scattering events. The net sum of the corresponding phonon energies that enter/exit at the hot and cold contact junctions gives the average phonon energy flux (net thermal flow) of np-Si that is then used to determine its thermal conductivity. The detailed explanation of this simulation procedure is systematically described in our previous works [43 - 47]. The contribution of optical phonons was assumed to be negligible as they have low $v_g(q)$ [39, 40, 42, 51, 52]. The basic porous geometry used in our simulations is illustrated in FIG. 5b. As given by previous works, all phonons that

impinge on a pore boundary scatter, and none are allowed to transmit [45, 48]. When a phonon is reflected, then a random number, that depends on the roughness of the pore domain, labeled as p , governs specular or diffuse scattering [38]. As in previous works [43 – 46, 48, 49], we do not assume that the phonon changes its energy at the interface as is common practice, but only its direction. The factor p ranges from 0 to 1, with $p = 0$ indicating purely diffusive and randomized reflection angle and with $p = 1$ indicating purely specular reflection where the angle of incidence is equal to the angle of reflection (see FIG. 5b). In our case we use $p = 0.1$ corresponding to a roughness $\Delta_{rms} \sim 0.3$ nm, which has been shown to correspond well to rough Si surfaces [43], and agrees well with our experimental observations, as outlined in the results section below.

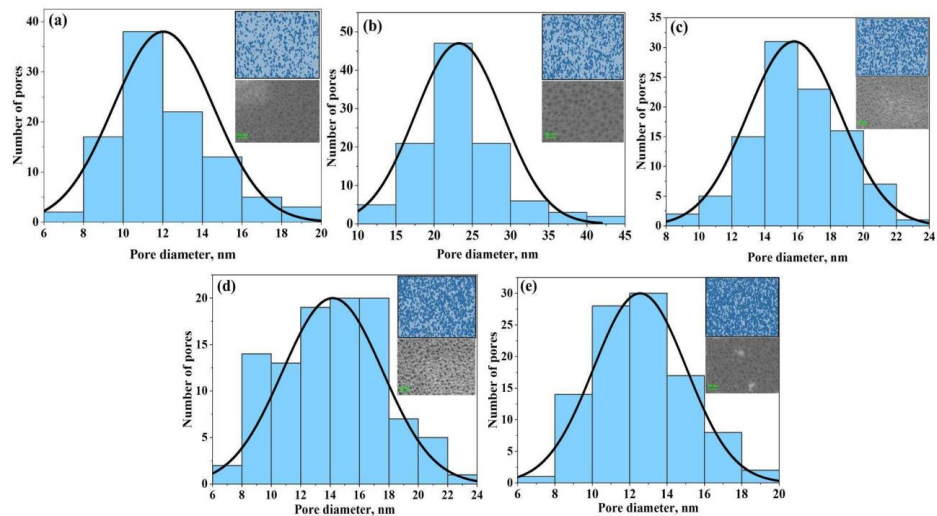


FIG. 6. Top-plane SEM images (insets) with corresponding image processing yielding Gaussian pore size distributions and pore areas of np-Si samples having different ϕ s measured by gravimetry: (a) 30%, (b) 40%, (c) 50%, (d) 60% and (e) 70%.

In the case of our porous geometries, the pores are defined as regions of zero transmission within a c-Si matrix, as implemented previously using the same MC methodology for porous Si media with $\phi \leq 50\%$ [43, 44, 48, 49] and even with $\phi = 70\%$ [52]. More importantly, the uncertainty of ± 0.2 W/mK in the thermal conductivity of np-Si with $\phi = 70\%$ computed by MC method is an order of magnitude larger than the heat conductivity of the air itself (0.03 W/mK) [53], which justifies the use of zero phonon transmission through air pores.

In the simulated schematics (FIG. 5a) the porous regions are given in white, while the c-Si phase regions are given in blue. The pores are randomized in both position and size, as encountered in realistic np-Si media. The development of these porous geometries with randomized pores is already well examined in our previous works [43 - 45]. The position distribution of the pores was taken to be randomly distributed, as in previous works in the literature [43, 44, 48]. Additionally, in this work we use the top-plane SEM images (FIG. 6) of the np-Si materials to determine the

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size distribution of pore diameters D . This was then used to inform the size distribution of the simulated pores.

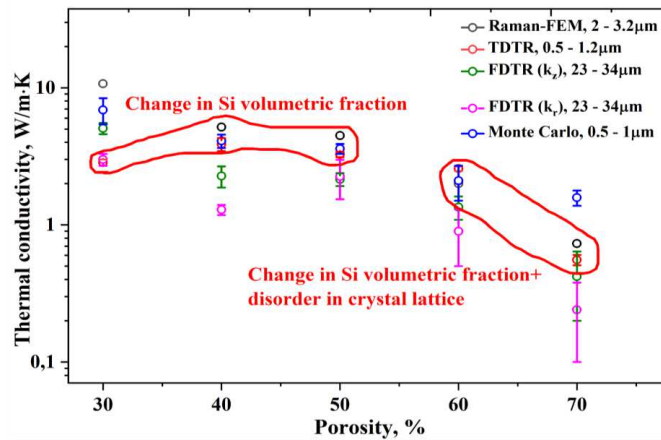


FIG. 7. TDTR, FDTR, Raman measurement and Monte-Carlo simulation results for the porosity-dependent thermal conductivity of np-Si.

Near-surface conductivities assessed by Raman and TDTR techniques were found to be in general agreement with conductivity computed by Monte-Carlo modeling, as seen in FIG. 7. At the same time, the difference between micron-scale depth-sensitive in-plane and cross-plane conductivity values measured by FDTR and those calculated by MC method is larger. This discrepancy could be explained by structural inhomogeneity of np-Si thin film samples taking place over thermally probed depths down to 23 - 34 μm , which is not accounted for in Monte-Carlo methodology for which computation domain = 0.5 - 1 μm .

In conclusion, we presented a multi-scale thermal depth profiling characterization across thick np-Si films with $\phi = 30 - 70\%$. The results from micro-Raman thermometry combined with FEM simulations indicate that the k of np-Si decreases with increasing ϕ , suggesting a lower volume fraction of c-Si content. Near-surface TDTR measurements, along with RBS results, reveal that the reduction in k is additionally influenced by the disordering of the crystal structure at high ϕ . Our k values measured by Raman and TDTR optical techniques are in general agreement with those obtained by semi-classical Monte-Carlo phonon transport simulations treating configurations of disordered np-Si architectures with various ϕ and D distributions. Furthermore, FDTR measurements demonstrate that np-Si becomes more thermally insulative at deeper micron-scale depths due to rise of ϕ as evidenced from cross-sectional SEM imaging, structurally inhomogeneous and thermally anisotropic behavior ($\eta \sim 0.6$). We envision applications of these porous semiconductors in advanced thermoelectric and thermal management devices where heat conduction needs be effectively controlled at various nano- and micro-scale depths.

Supplementary material

See the **Supplementary Material** for detailed information about the np-Si fabrication process and structural characterization measurements, Monte-Carlo simulation details, Raman, Ellipsometry, TDTR and FDTR measurement conditions.

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Conflict of interest

The authors declare no conflict of interest.

Data availability

The raw data required to reproduce these findings are available from the corresponding author, upon email request. The processed data required to reproduce these findings are also available upon request.

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