Supplemental Information:

Noble-Gas -Infused Neoprene Closed-Cell Foams Achieving Ultra-Low Thermal Conductivity Fabrics

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Porosity Determination for Unmodified Neoprene

The porosity of unmodified neoprene foam was determined via image analysis in ImageJ of the scanning electron micrograph (SEM) images shown in Fig. S1a and b. The SEM image shown in Fig. S1a was converted to binary and partitioned into six regions of interest, as shown in Figure S1(b). Using the plugin BoneJ, the percent volume fraction of black pixels (gas) to white pixels (neoprene rubber) was determined for each region of interest: 83.5%, 83.8%, 80.0%, 86.0%, 84.6%, 82.6%. These values correspond to estimates of the porosity for the neoprene foam. The overall porosity of the foam was reported as the mean with an error corresponding to the standard error of the mean (95% confidence interval): 83±2%.



Fig. S1: (a) Scanning electron micrograph (SEM) image of unmodified neoprene. (b) SEM image from a) converted to binary in ImageJ in order to estimate the porosity of the neoprene foam. The dashed yellow lines indicate the six regions of interest used to calculate six values of porosity for the foam.

Estimation of Neoprene Rubber Density

If we assume the neoprene foam to have a porosity of approximately 83%, we can use the definition of porosity, as given previously by Bardy et al.¹, to estimate the rubber density:

$$\phi = 1 - \frac{\rho_{foam}}{\rho_{rubber}} \tag{S1}$$

Here, ϕ is the porosity, ρ_{foam} is the density of the neoprene foam, and ρ_{rubber} is the density of the solid neoprene rubber component. The foam density is computed by dividing the measured mass of a neoprene disc (1.353 g) by the volume of the disc, which depends on time according to the measured values of radius and thickness. For a standard untreated neoprene disc, the radius and thickness are approximately 2.5 cm and 0.614 cm, respectively. Approximating the neoprene disc as a cylinder, the volume is determined to be 15.35 cm³, implying a density of 0.088 g/cm³. Solving equation (S1) for the rubber density, we have $\rho_{rubber} = 0.51849 \text{ g/cm}^3$. However, recalling that there is a 2% uncertainty in the porosity estimate, this uncertainty must propagate to an associated uncertainty in the rubber density. Calculating the upper and lower bounds for the density based on the appropriate bounds on the porosity, we have

$$0.464 < \rho_{rubber} < 0.588 \text{ g/cm}^3$$
. (S2)

These limits for the rubber density are used to determine the bounds of the predicted range of simulated values in Fig. 3c in the main text.

Estimation of Neoprene Rubber Thermal Conductivity

Given that the porosity and thermal conductivity of untreated neoprene foam are approximately 83% and 0.051 W/m/K respectively, we can use Maxwell's relation (equation 1 in the main text) to estimate the thermal conductivity of the rubber component. A simple iterative solution procedure yields **0.228** W/m/K for the rubber thermal conductivity.

While this value lies outside the range reported in the literature for the thermal conductivity of neoprene rubber¹ (0.11 - 0.19 W/m/K), we have measured larger values for neoprene rubber. Three measurements were taken (0.4587, 0.4561, and 0.4509 W/m/K), for an average of **0.455 W/m/K** and a standard deviation of **0.00397 W/m/K**.

Thus, our assumed value for the rubber thermal conductivity lies within a realistic range.

Modification Process for Neoprene Foam Samples

A schematic for the charging process for the neoprene foam "bare" samples is shown in Fig. S2. Neoprene foam samples are placed in a 1.9 liter tank at a pressure of 20 psig of the charging gas (argon, krypton, or xenon) for a period of 5 to 7 days.



Fig. S2: Process flow for charging neoprene foam samples with xenon, argon, or krypton gas.

SEMs of Modified Neoprene Foams

SEM images of argon and xenon charged neoprene foam are shown in Fig. S3a and b, respectively. The SEM images can be compared with Fig. 1a, and suggest the absence of damage to the neoprene foams due to the high molecular weight noble gas infusion process.



Fig. S3: (a) SEM image of a neoprene foam sample following charging with argon gas. (b) SEM image of a neoprene foam sample following charging with xenon gas.

Theory and Accuracy of Hot Disk Method for Thermal Conductivity

Theory

The Transient Plane Source (TPS)² is an ISO standard transient measurement technique³ for determining the thermal conductivity, thermal diffusivity, and heat capacity polymeric materials, which has been used to determine the thermal conductivity of a variety of polymers^{4,5} and insulating materials^{6,7}.

The standard TPS measurement technique employs a plane source heating element, which also acts as a proxy for temperature measurement. The method assumes that the plane source is immersed in an infinite medium, and the relevant equations are shown below.

$$\Delta T(t) = \frac{P_0}{\frac{3}{\pi^2 ak}} f(t^*)$$
(S3)

$$f(t^*) = \frac{1}{n^2(n+1)^2} \int_0^t \frac{1}{s^2} \sum_{l=1}^n l \sum_{j=1}^n j \exp\left(-\frac{\left(\frac{j}{n}\right)^2 + \left(\frac{l}{n}\right)^2}{4s^2}\right) I_0\left(\frac{\left(\frac{j}{n}\right)\left(\frac{l}{n}\right)}{2s^2}\right) ds$$
(S4)

where $\Delta T(t)$ refers to the temperature increase of the sensor, P_0 refers to the power input of the sensor, *a* refers to the radius of the sensor, *k* refers to the thermal conductivity of the sample, *t* refers to time, t^* refers to a dimensionless time, *n* refers to the number of concentric rings in the sensor, and I_0 refers to the modified Bessel function.

As can be seen from equation (S3), a plot of $\Delta \overline{T}(t)$ vs. $f(t^*)$ should result in a linear relation with the slope being related to the thermal conductivity of the sample.

Estimation of Accuracy

We investigated the accuracy of the transient plane source technique for measuring the thermal conductivity of our neoprene samples by measuring the thermal conductivity of a NIST standard reference material: expanded polystyrene board (SRM 1453)⁸. The certified thermal conductivity of the unit is given by equation (S5).

$$k = 0.00111 - 0.0000424\rho + 0.000115T \tag{S5}$$

where k is the thermal conductivity (W/m-K), ρ is the density (kg/m³), and T is the temperature (K), which is valid from 281 K to 313 K.

We measured an average thermal conductivity of 0.03551 W/m-K with a standard deviation of 0.00009 W/m-K from five measurements at a temperature of 294 K with our transient plane source device. We used the same settings as for our neoprene samples ($P_0 = 15 \text{ mW}$ for 80 seconds). The average density of the expanded polystyrene board is 41.5 kg/m³, and from equation (S5), the expected thermal conductivity is 0.03316 W/m-K. Thus, we calculate an error of approximately 7% for our measurements.

Heat Transfer Image Import Model for Initial Thermal Conductivity

To better understand the active mechanism of heat transfer in neoprene foam, we simulated the process using COMSOL Multiphysics. The neoprene microstructure was captured by importing a grayscale optical micrograph of neoprene foam into COMSOL. The image intensity is mapped to a dimensionless function ranging from 0 to 1, im1(x,y), such that im1 = 0 corresponds to pure black and im1 = 1 corresponds to pure white. We then defined a spatially-dependent thermal conductivity, $k_{local}(x,y)$, by

$$k_{local}(x,y) = \begin{cases} k_{rubber} & \text{if } \operatorname{im1}(x,y) > \beta \\ k_{qas} & \text{if } \operatorname{im1}(x,y) < \beta \end{cases}$$
(S6)

where the fitting parameter β is a cutoff value of the image intensity to be defined.

We then conduct simulations of steady-state heat transfer in the neoprene piece. The boundary conditions are a constant temperature T_H on the inside of the neoprene and T_C on the outside. The output data of interest from the simulations is the volume-average heat flux $\overline{q_{neo,x}}$ (in the horizontal, i.e. *x*-direction) through the neoprene (which is independent of T_H and T_C). The effective thermal conductivity k_{eff} of the neoprene is then determined from Fourier's Law of Heat Conduction,

$$k_{eff} = \left| \frac{\overline{q_{neo,x}}}{\left(T_{H} - T_{C}\right)} \right|.$$
(S7)

Here δ_{neo} is the thickness of the neoprene foam and T_H and T_c are the temperatures on the immediate inside and outside of the foam, respectively.



Fig. S4: (left) The dimensionless image intensity function, im1, for an imported image of neoprene foam. For regions where im1 is below a certain threshold, we assign the thermal conductivity of the filling gas of interest to that region; for regions where im1 exceeds the threshold we assign the rubber thermal conductivity. (right) the local thermal conductivity, k_{local} , for the same neoprene image shown at left. For this case, the pores are assumed to be filled with xenon.

If we assume β = 0.41, we obtain the effective thermal conductivities for the control and gas-infused neoprene samples displayed in Table S1.

Table S1: Comparison of simulated and measured thermal conductivities of neoprene foams.

Fill Gas	Therm cond. (Simulation) [W/(m·K)]	Therm cond. (Experiment) [W/(m·K)]
Air	0.05686	0.05701
Ar	0.04887	0.04268
Xe	0.03419	0.03515

Kr 0.03581 0.03280

Although the agreement is generally good between the simulations and experiments, some error may have been introduced by the use of a 2-D simulation to represent an inherently 3-D porous network. Still, it is notable that the agreement is this good assuming a 2-D microstructure.

We emphasize that these simulations assume heat transfer occurs only by conduction in both the gas and rubber phases. The gas thermal conductivity is set to the pure-gas value for the gas under consideration. Thus, the results of these simulations motivate two important conclusions: (1) Heat transfer occurs in the neoprene essentially by pure conduction through the gas and rubber phases; (2) the filling gas completely replaces the air that originally filled the pores.

Permeation Experiments to Measure Gas Diffusivity in Neoprene

To determine the effective gas diffusivities in the neoprene foam, we measured the rate of Ar and Xe permeation through the neoprene using a custom-built gas permeation module, as illustrated in Fig. S5a (additional details in experimental section). After the neoprene foam was sealed inside the membrane module, a feed of pure Ar or Xe was flowed over the upstream side of the membrane at a gauge pressure of 445 Torr, while the downstream side of the membrane was continually swept with N₂ gas at near atmospheric pressure. Due to the pressure differential across the neoprene, over time, the Ar and Xe gas permeated through the foam, and the permeate gas was carried by the N₂ sweep gas into an online mass spectrometer configured for real-time gas analysis.

The results of these permeation experiments are shown in Fig. S5b and c. The counts detected by the mass spectrometer are directly proportional to the instantaneous flux of the Ar or Xe through the neoprene. At the beginning of each measurement, the flux is low and increasing as the Ar and Xe diffuse into the foam and build up a concentration gradient. Eventually the flux reaches a steady state value, as expected. The time required to reach the steady state flux depends on the effective diffusivity of the gas in the neoprene. Fitting the mass spectrometer data to a model enables us to extract the effective diffusivities of the Ar and Xe, as discussed below.



Fig. S5: (a) Apparatus for measuring gas permeation through neoprene foam. Active area of the neoprene membrane is 6 cm² and the thickness of the neoprene membrane is 1.6 mm. (b) Experimentally measured mass spectrometer counts for argon as a function of time (symbols), along with a best-fit of equation (S8). From this curve fitting, we can extract an effective diffusivity of 1.9×10⁻¹⁰ m²/s for argon in neoprene foam. (c) Mass spectrometer counts for xenon as a function of time (symbols) with fit of equation (S8). From this analysis, the effective diffusivity for xenon in neoprene foam is determined to be 3.967×10⁻¹⁰ m²/s. The experimental data in both panels (b) and (c) has been smoothed using the "smooth" function in MATLAB, which uses a moving average filter with a span of 11 data points.

For this experimental setup, gas diffusion may be modeled as unidirectional. Under this assumption, it is straightforward to show^{9,10} that the number of counts recorded by the mass spectrometer, Q(t), should be well-described by the analytical solution to the 1-D diffusion equation,

$$Q(t) = Q_0 \left[1 + 2\sum_{n=1}^{\infty} (-1)^n \exp\left\{ -(n\pi)^2 \frac{D_{foam}t}{d^2} \right\} \right],$$
 (S8)

where Q_0 is the steady-state count rate, D_{foam} is the (initially unknown) effective diffusivity of the gas in the neoprene foam, and *d* is the thickness of the foam sample. By fitting equation (S8) to the measured counts, D_{foam} can be determined for the permeating gas. The data plotted in Fig. S5b and c was produced by applying to the oversampled raw data a moving average filter with a span of 11 data points, using the "smooth" command in MATLAB. There are two free parameters in the curve-fitting process. The first is D_{foam} , and the second is the amount of time Δt by which the fit curves in Fig. S5b and c were shifted to account for the time between when the gas flow was initiated and when data was first recorded. Using least-squares curve fitting, we obtain the values of D_{foam} and Δt for Ar and Xe in neoprene foam that are shown in Table S2 below.

Table S2: Results of curve fitting of equation (S8) to the neoprene permeation data for Ar and Xe. Here Δt is the time shift that is artificially applied to the experimental data to optimize the quality of the fit (as determined by least-squares fitting of the fit curve, equation (S8), to the experimental data.

Gas	<i>D_{foam}</i> (×10 ⁻¹⁰ m ² /s)	Δt (min)
Ar	1.9029	13.240
Хе	3.9670	3.718

The solubility of a gas in a polymer membrane is defined as the amount of gas that dissolves in the membrane per unit volume of the solid material per unit partial pressure of the gas above the membrane. The most common units for solubility are cm³_{STP}/cm³-Pa. The solubilities of argon and nitrogen in neoprene rubber (polychloroprene) were measured by Barrer¹¹. However, these values are not directly applicable to neoprene foam. Since we are modeling the neoprene foam as a homogeneous material, it is necessary to assign an effective solubility for the foam as a whole.

Since the foam by definition includes gas and solid phases, the effective foam solubility must take into account both gas stored in the gas cells and in the solid rubber. Let us define the effective foam solubility, S_{foam} , as the amount of gas molecules contained in a given volume of the foam (assuming a constant porosity) per unit partial pressure of the gas above the foam. If the insulating gas is modeled as an ideal gas, it can be shown that S_{foam} is related to the conventional solubility, S, by

$$S_{foam} = \frac{\phi}{RT} + S(1 - \phi).$$
(S9)

The first term in equation (S9) represents gas that is stored in the gas cells of the neoprene; the second term represents gas molecules that have dissolved in the solid rubber. This equation is derived assuming the system is at equilibrium, so that the gas pressure inside the cells is equal to the pressure of the gas above the neoprene foam. In this form of the equation and in the rest of this paper, the units of S_{foam} and S are both mol/m³-Pa. Thus, S_{foam} may be thought of as the concentration of gas in the foam (in mol/m³) per unit partial pressure of the gas above the foam (in Pa).

Thermal Conductivity Evolution in Different Environments

Photographs for the experimental setups for storing the argon-charged neoprene samples in stagnant and stirred water are shown in Fig. S6a and b, respectively. The thermal conductivities of the samples were measured in an air environment, and in between measurements the samples were either stored in the apparatus shown in Fig. S6a or b.



Fig. S6: (a) Photo of the argon-charged neoprene samples stored in a closed container with stagnant deionized water. (b) Photo of the experimental setup for storing argon—charged neoprene samples in an open container with deionized water stirred at 500 RPM.

Data for the argon-infused neoprene samples stored in stirred water, stagnant water, and air are shown in Fig. S7. The leakage rate (discharge) of the argon from the bare neoprene foams is significantly reduced for samples stored in both stagnant and stirred water, as compared with those stored in air.



Figure S7: Thermal conductivity vs. time for bare neoprene charged with argon and stored in air and neoprene samples charged with argon and stored in stagnant (green triangles) and stirred water (blue circles) in between measurements. This figure indicates that leakage of the insulating gas from the neoprene occurs more slowly in stirred water (simulating a swimming diver) than in air and is slowest in stagnant water.

The raw experimental thermal conductivities for the argon-charged samples stored in the stirred deionized water are shown in Fig. S8a. The red circle indicates a suspected outlier. We fit the data to a linear regression and evaluated the standardized residuals, as shown in Fig. S8b. From the standardized

residual, we determined that the data point occurring at approximately 70 hours is most likely an outlier, as it exists almost three standard deviations from the linear fit.



Figure S8: (a) Measured thermal conductivity as a function of time for argon-charged neoprene samples stored in stirred deionized water, as shown in Fig. S7. The data is fit to a linear regression, and the linear fit for the data is shown in the plot. The suspected outlier is indicated by a red circle. (b) The standardized residuals for the linear fit in a). The suspected outlier is indicated by the red circle and occurs almost three standard deviations from the fit.

Radius of Neoprene Foams vs. Time

Fig. S9 shows the evolution of the radius of an argon-infused disc of neoprene foam as a function of time after removal from the argon environment. The radius is observed to decay slightly, from 2.50 to approximately 2.46 cm. The dynamics of gas leakage from the neoprene are not affected significantly by the change in volume induced by the change in radius. However, even a slight change in volume does produce a noticeable change in the porosity of the neoprene, to which the overall thermal conductivity is rather sensitive (as reflected in equation (1) in the main text).



Figure S9: Radius of argon-infused neoprene disc (measured using optical images captured with USB camera and image analysis using ImageJ software) as a function of time after removal from the gas environment. Red circles are experimental measurements and blue curve is a fit of the form $r_{fit} = ae^{-bt} + c$. Here the constants *a*, *b*, and *c* are 0.0357 cm, -0.1897 hr⁻¹, and 2.4605 cm, respectively.

Initial and Boundary Conditions for Numerical Gas Diffusion Model

The shrinkage and expansion of the neoprene foam during charging and discharging are critical to track precisely, since they affect the porosity of the foam, on which its thermal conductivity depends strongly, as dictated by Maxwell's result. In this work, we measure the variation of both radius and thickness with time by capturing high-resolution images of neoprene samples during the Ar discharge process. Fig. 3b shows the measured values of thickness versus time for an argon-infused neoprene disc; these thickness measurements are direct inputs to the discharging simulations. For the charging simulations, such a direct measurement of thickness or radius at regular intervals is not possible because the neoprene is contained inside a closed, opaque container. In this case, the initial and final neoprene thicknesses are measured, and for simplicity a linear increase of thickness with time is assumed. In both the charging and discharging simulations, we use the moving-mesh boundary conditions to track the deformation of the neoprene disc.

The mass transfer boundary conditions for N_2 and Ar are different for the charging and discharging simulations. For the discharging case, we ignore gas flux through the top and bottom surfaces of the neoprene disc (since neither of these surfaces is exposed to the background air). Instead, we assume Henry's Law applies on the outer surface of the neoprene disc only, and vanishing flux through the top and bottom surfaces. In this case, the concentration of Ar vanishes at the outer edge and the concentration of nitrogen is equal to the foam solubility of nitrogen multiplied by the partial pressure of nitrogen (assumed to be atmospheric):

$$C_{Ar} = 0, \ C_{N2} = S_{foam,N2}P_{N2}, \qquad \text{Outer edge surface}$$

$$\mathbf{n} \cdot \mathbf{j}_{Ar} = 0, \ \mathbf{n} \cdot \mathbf{j}_{N2} = 0, \qquad \text{Top and bottom surfaces}$$
(S10)

The boundary conditions described by equation (S10) are visualized in Fig. S10a. For the charging case, the neoprene disc is assumed to be lying flat on the bottom of the charging container. Thus, we ignore the flux of gas into the bottom surface of the disc. On the top and outer edge surfaces of the disc, we assume that the concentration of nitrogen is zero and that of the argon is equal to the foam solubility multiplied by the partial pressure of argon (20 PSIG). In other words, we assume equilibrium (dictated by Henry's Law) applies at all times on the top and outer edge surfaces:

$$C_{N2} = 0, C_{Ar} = S_{foam,Ar} P_{Ar}, \text{ Top surface and outer edge surface}$$

$$\mathbf{n} \cdot \mathbf{j}_{Ar} = 0, \quad \mathbf{n} \cdot \mathbf{j}_{N2} = 0, \text{ Bottom surface}$$
(S11)

The boundary conditions described by equation (S11) are visualized in Fig. S10b.

For the discharging simulations, we assume that the initial concentration of nitrogen in the neoprene is zero and the concentration of argon is the foam solubility of argon multiplied by the filling pressure of argon ($P_{Ar} = 2.4$ atm),

$$C_{N2}(r, z, t = 0) = 0,$$

$$C_{Ar}(r, z, t = 0) = S_{foam, Ar} P_{Ar}.$$
(S12)

For the charging simulations, the initial concentration of argon in the neoprene is zero and the initial concentration of nitrogen is equal to the effective foam solubility (derived above) multiplied by atmospheric pressure:

$$C_{Ar}(r, z, t = 0) = 0,$$

$$C_{N2}(r, z, t = 0) = S_{foam, N2}P_{atm}.$$
(S13)



Figure S10: (a) Schematic of simulation domain showing dimensions, initial conditions, and boundary conditions assumed in the simulation. (b) Schematic of charging simulations showing boundary and initial conditions. Gas flux through the bottom surface is ignored because the neoprene disc is assumed to sit flat on the bottom of the charging container.

Material Application for Low Temperature Wetsuits

The process for infusing an entire wetsuit with either krypton or argon is shown in Fig. S11a. The process is similar to that shown in Fig. S2, except the tank size is 19 L. Data for an argon-infused wetsuit compared with bare neoprene is shown in Fig. S11b. The thermal conductivities for controls and constituent materials of the composite are also provided.



Figure S11: (a) Photograph and schematic of apparatus for charging an entire wetsuit with insulating gas. An entire men's medium wetsuit can fit easily into a 19 L charging tank. (b) Thermal conductivity vs. time for bare neoprene charged with argon compared with data for a full wetsuit, also charged with argon.

Thermal Conductivity of Ar-Infused Neoprene Foam at Low Temperatures in an Aquatic Environment

We investigated the performance of argon-infused neoprene in an aquatic environment at 5 °C and compared its performance with both unaltered and argon-infused neoprene at room temperature under

air exposure. The results of the study are shown in Fig. S12. The aquatic environment consisted of a tank of tap water held at 5 °C using a thermoelectric heat bath (Echotherm chilling/heating dry bath) for the entirety of the measurements. The samples were not removed during the course of the measurements. In addition, the neoprene samples were stacked and tested with the same thermal conductivity measurement protocol as reported in the methods section.



Figure S12: Thermal conductivity as a function of time for unaltered neoprene and argon-infused neoprene under room temperature (RT) air exposure and 5 °C water exposure.

Effect of Elevated Diffusivity on Gas Charging

The charging process can be accelerated by increasing the temperature of the insulating gas.

Generally speaking, the charging time is governed by the *permeability* of the membrane to the gas of interest. It is well-known that the permeability is the product of the diffusivity and solubility of the gas of interest ¹²:

$$P = SD. \tag{S14}$$

Studies of gas permeation through solid polymer membranes have found that the diffusivity of a gas in a rubber material is the most sensitive parameter to temperature ¹³, and solubility and permeability are relatively insensitive to temperature. Thus the main effect of increased temperature on the gas transport will be to increase the diffusion coefficient, which for gases follows an Arrhenius dependence

$$D = D_0 \exp\left(\frac{E_A}{RT}\right)$$
(S15)

where D_0 is a pre-exponential factor, E_A is an activation energy for diffusion, and R is the gas constant. This equation governs the response of the diffusivity of the gas in the *solid rubber* material.

To get a sense for the dependence of charging time on diffusivity, we conducted simulations of gas charging of neoprene foam with the diffusivities of both gases scaled by factors of 2 and 5. The results are depicted below.



Figure S13: Thermal conductivity vs. time for charging under elevated temperature conditions. In the green and magenta lines, the diffusion coefficients of both Ar and N₂ have been scaled by the indicated factors. This figure suggests that charging the neoprene at elevated temperatures could lead to reductions in the charging time.

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