Fabrication of 3D Macroporous Nanoelectronic Networks. Key steps used in the fabrication of the 3D macroporous nanowire nanoelectronic networks were as follows (SI Text): (i) Lithography and thermal deposition were used to pattern a 100-nm nickel metal layer, where the nickel served as the final relief layer for the 2D free-standing macroporous nanowire nanoelectronic networks. (ii) A 300- to 500-nm layer of SU-8 photoresist (2000.5; MicroChem Corp.) was deposited over the entire chip, followed by prebaking at 65 °C and 95 °C for 2 and 4 min, respectively, then (iii) the synthesized nanowires were directly printed from growth wafer over the SU-8 layer by the contact printing. Lithography (photolithography or electron beam lithography) was used to define regular patterns on the SU-8. After postbaking (65 °C and 95 °C for 2 and 4 min, respectively), SU-8 developer (MicroChem Corp.) was used to develop the SU-8 pattern. Those areas exposed to UV light or electron beam became dissolvable to SU-8 developer, and other areas were dissolved by SU-8 developer. Those nanowires on the nonexposed area will be removed by further washing away in isopropanol solution (30 s) twice, leaving those selected nanowires on the regular pattern SU-8 structure. The SU-8 patterns were cured at 180 °C for 20 min. (iv) A 300- to 500-nm layer of SU-8 photoresist was deposited over the entire chip followed by prebaking at 65 °C and 95 °C for 2 and 4 min, respectively. Then lithography was used to pattern the bottom SU-8 layer for passivating and supporting the whole device structure. The structure was postbaked, developed, and cured by the same procedure as described above. (v) Lithography and thermal deposition were used to define and deposit the metal contact to address each nanowire device and form interconnections to the input/output pads for the array. For the mesh device, in which the metal is nonstressed, symmetrical Cr/Pd/Cr (1.5/50–80/1.5 nm) metal was sequentially deposited followed by metal liftoff in acetone. For the self-organized networks, in which the metal is stressed, nonsymmetrical Cr/Pd/Cr (1.5/50–80/50–80 nm) metal was sequentially deposited followed by metal liftoff in acetone. The tip of the AFM was placed down (loading) and then up (unloading), with typical data measuring the thermal vibration spectrum (3).

Bending Stiffness Analysis. Due to the residual stress, The SU-8/metal/SU-8 elements bend upward from the substrate (due to
internal stress of the asymmetric metal layers) with a constant curvature, $K_0$, and projected length, $l$, where $l_0$ is the free length defined by fabrication. We use the curvilinear coordinate $s$ to describe the distance along the curved ribbon from the fixed end, and the coordinate $x$ to describe the projection position of each material point of the ribbon (Fig. S3A). For a specific material point with distance $s$, the projection position $x$ can be calculated as $x = f \cos (\varphi) ds$, where $\varphi = K_0 s$ is the angle between the tangential direction of the curvilinear coordinate $s$ and the horizontal direction (Fig. S3B). Integration yields $x = \sin (K_0 s)/K_0$ and when $x=0$ and $s=0$, $K_0 = 0.0128 \text{mm}^{-1}$ for typical experimental parameters $l_0 = 100 \text{mm}$ and $l = 75 \text{mm}$.

Because the element is deflected a distance $d$ by the AFM tip with a force $F$, each material point is rotated by an angle, $\varphi$ (Fig. S3B), where the anticlockwise direction is defined as positive. Assuming a linear constitutive relation between the moment $M$ and curvature change $d\varphi/ds$ (4) yields

$$\frac{d\varphi}{ds} = \frac{M}{wD_m}$$

[S2]

where $M$ is the moment as a function of position, $x$ (Fig. S3), and $w$ is the width.

$$M(x) = -F(l-x)$$

[S3]

Solving for the bending stiffness, $D_m$, with the assumption that $\varphi$ is small so that $\sin \varphi \approx \varphi$ yields

$$D_m = \frac{F}{wd} \left( \frac{l}{K_0} \sin (K_0 l_0) + \frac{1}{K_0^2} \left( l \cos (K_0 l_0) - l + l_0 \right) \right)$$

$$+ \frac{1}{K_0^3} \left( \frac{\sin (2K_0 l_0)}{4} - \sin (K_0 l_0) \right).$$

[S4]

The slope of a representative loading force-deflection curve yields $F/d = 12 \text{nN}/\mu \text{m}$ (Fig. S2C), and using Eq. S4, the calculated bending stiffness per width ($w = 5 \mu \text{m}$) is $D_m = 0.358 \text{nN/m}$. For typical 3D macroporous nanoelectronic networks, the area fraction for both types of elements (i.e., SU-8 and SU-8/metal/SU-8) can range from 1% to 10%, yielding values of the effective bending stiffness from 0.0038 to 0.0378 nN/m.

Three-Dimensional Macroporous Chemical Sensors. Agarose (Sigma) was dissolved into DI water, made as 0.5%, and heated up to 100 °C. The gel was drop-cast onto the device and cooled to room temperature. DAPI (Sigma) was used to dope the gel for the confocal 3D reconstructed imaging. A microfluidic polydimethylsiloxane (PDMS) fluidic chamber with input/output tubing and Ag/AgCl electrodes was sealed with the silicon substrate and the device or device/gel hybrid using silicone elastomer glue (Kwik-Sil; World Precision Instruments, Inc.). Fresh medium was delivered to the device region through both inner and outer tubing. The solution pH was varied stepwise inside the channel by flowing (20 mL/h) 1× phosphate buffered solutions with fixed pH values from pH 6 to 8. The recorded device signals were filtered with a bandpass filter of 0–300 Hz.

Three-Dimensional Macroporous Nanoelectronic/Elastomer Strain Sensors. A freestanding 2D macroporous nanoelectronic network was suspended in water and placed on a thin (200–500 μm) piece of cured silicone elastomer sheet (Syngard 184; Dow Corning). The hybrid macroporous nanowire network/silicon elastomer was rolled into a cylinder, infiltrated with uncured silicone elastomer under vacuum, and cured at 70 °C for 4 h. The resulting hybrid nanoelectronic/elastomer cylinders had volumes of ~300 μm³ with volume ratio of device to elastomer of <0.1%. The structure of the macroporous electronics/elastomer hybrid was determined using a HMXST X-ray micro-CT system with a standard horizontal imaging axis cabinet (HMXST225; Nikon Metrology, Inc.). In a typical imaging experiment, the acceleration voltage was 60–70 kV, the electron beam current was 130–150 mA, and no filter was used. BGStudio MAX (version 2.0; Volume Graphics GmbH) was used for 3D reconstruction and analysis of the micro-CT images, which resolve the 3D metal interconnect structure and nanowire source/drain (S/D) contacts; the Si nanowires were not resolved in these images but were localized at the scale of the S/D contacts. The piezoelectric response to the strain of the nanowire devices was calibrated using a mechanical clamp device under tensile strain (Fig. S5), where the strain was calculated from the length change of the cylindrical hybrid structure. The bending strain field was determined in experiments where the cylindrical hybrid structure, with calibrated nanowire strain sensors, was subject to random bending deflections.

Fig. S1. Interface to macroporous nanoelectronic network. Zoom-in of the region enclosed by the white dashed box in Fig. 2D. The white arrows highlight several wire bonds between the printed circuit board connector board and metal pads on the edge of the macroporous nanoelectronic network.
Fig. S2. Bending stiffness measurements. (A) Schematic illustrating the measurement of the bending stiffness of a representative SU-8/metal/SU-8 element in the macroporous nanoelectronic networks. Electron beam lithography is used to define substrate-fixed and substrate-free beams, where internal stress in the central metal layer causes the structure to bend up upon relief from the substrate (SI Text). The tip of the AFM is placed at the free end of the ribbon, and then translated vertically downward (loading) and upward (unloading) to yield the force-displacement curves. In the scheme: \( w \), the width of the ribbon; \( l_0 \), the length of the ribbon; \( l \), the projected length of the ribbon; and \( d \), the displacement of the AFM tip. (B) Optical micrograph of the fabricated structural element, where the substrate-fixed portion is highlighted by the red dashed rectangle, and the free beam is in the upper portion of the image with a width of 5 \( \mu \)m and a length of 100 \( \mu \)m. (C) A typical force-displacement curve with \( F/d \) for loading and unloading of 12 and 10.5 nN/\( \mu \)m, respectively. Similar deviation between the loading and unloading has been attributed to inelastic deformation (1, 2); hence, we use the larger loading value in calculations to provide an upper limit.


Fig. S3. Schematics for calculations. (A) A schematic of the position of the substrate-free beam before (black) and after (red) applying a calibrated force, \( F \), and vertical displacement, \( d \), at the end of the beam with the AFM. (B) The angle between the tangential direction of a material point on the beam and the horizontal direction, \( \psi \), of the ribbon before (black) and after displacement, \( \psi + \varphi \), (red). \( l_0 \), total length of the ribbon; \( l \), projection of the ribbon.
Fig. S4. Localization of 3D macroporous nanoelectronic devices. The 3D macroporous nanoelectronic FET devices exhibit photoconductivity (1) that was used to determine spatial positions using a confocal microscope equipped with an analog signal input box (Materials and Methods). (A) Schematic of photocurrent detection and correlation with confocal microscopy laser spot scanning position. A 405-nm laser wavelength, 100× water-immersion lens, and 0.1-mV source/drain device bias-voltage were used in the experiments. (B) High-resolution (1 nm per pixel) photocurrent image (I) from a single nanowire device (2-μm channel length between upper/lower metal contacts) recorded scanning in xy plane. The red dashed line indicates the direction perpendicular to the nanowire axis. The black dashed lines indicate the boundaries of metal contacts. (II) Photocurrent measured along the red dashed line in I. Experimental data are fit with a Gaussian distribution (red solid curve). (C) Distribution of the center point positions determined from 20 independent scans in the region indicated in Fig. 3 B, II and about the single scan line (red dashed line) shown in Fig. 3 B, I.

Fig. 55. Calibration of the 3D macroporous nanoelectronic strain sensors. (A) Conductance change vs. time as a 10% tensile strain was applied to hybrid 3D macroporous nanoelectronic networks/PDMS cylindrical sample. The downward- and upward-pointing arrows denote the times when the strain was applied and released, respectively. The direction of strain on the cylindrical hybrid sample and projected position of the macroporous nanoelectronic networks are indicated in the optical micrograph (Right). The conductance changes of 11 measured nanowire devices (labeled arbitrarily in terms of increasing sensitivity) were recorded and used for the conductance change per strain calibration. (B) Strain sensitivity calibration of the nanowire devices is plotted in 3D. The data points are color-coded by the sensitivity of the devices (A).