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(54) Title: METHODS AND SYSTEMS FOR WELDING OF SEMICONDUCTOR NANOWIRES

(57) Abstract: Methods and systems for capillarity-driven welding of semiconductor nanowires for flexible three-dimensional networks with ohmic interconnects. Methods of synthesizing electrical connections between semiconductor nanowires by capillarity-induced welding can comprise creating or providing nanowires, aligning the nanowires to yield inter-nanowire points-of-contacts, and welding the nanowires together at the inter-nanowire points-of-contact on each nanowire by capillarity-driven surface diffusion. Flexible and conductive thin nanowire films that have Ohmic junctions are also provided. Three-dimensional nanowire film materials, comprising a plurality of nanowires overlapping in a random orientation and welded together to form Ohmic junctions, are also provided.



DESCRIPTION

METHODS AND SYSTEMS FOR WELDING OF SEMICONDUCTOR NANOWIRES

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CROSS REFERENCE TO RELATED APPLICATIONS

This application claims the benefit of and priority to U.S. Provisional Patent Application Serial No. 62/357,071, filed June 30, 2016, the disclosure
10 of which is incorporated by reference herein in its entirety.

TECHNICAL FIELD

The presently disclosed subject matter is directed to methods and systems for capillarity-driven welding of semiconductor nanowires for flexible
15 three-dimensional networks with ohmic interconnects.

BACKGROUND

Nanowires (NWs) composed of or comprising semiconductors and metals have been widely developed for applications in electronics, photonics, energy, and biology.^{1,2} These applications are often demonstrated
20 using small, proof-of-concept systems; however, further advancements will require integration on a massive scale, connecting millions of NWs into device structures. To allow for these applications, substantial effort has been focused on NW directed-assembly³ and self-assembly methods, which
25 include flow-alignment⁴, mechanical transfer printing⁵, dip coating⁶, electric-field assisted placement⁷, and top-down patterning strategies⁸. These methods generally rely on conventional lithographic fabrication to form the electrical connections between NWs.

For metallic NWs, electrically-active networks have been developed as a class of transparent and conductive thin films with silver⁹ (Ag), copper¹⁰ (Cu), and gold¹¹ (Au) NWs, as well as carbon nanotubes¹². Ohmic connections
30 between these components have been formed through a variety of techniques¹³, including cold welding¹⁴, plasmonic welding,¹⁵ thermal

annealing¹⁶, mechanical pressure¹⁷, diffusion bonding¹⁸, and electron-beam induced welding¹⁹. In comparison to metallic NWs, however, semiconductor NWs could offer a wide range of more advanced functionality by encoding field-effect transistors²⁰, p-n junctions²¹, and memory bits²² within the individual
5 NWs of the network²³.

Flexible meshes of silicon (Si) and germanium (Ge) NWs have been fabricated²⁴⁻²⁸, but the structures are electrically-inactive or exhibit high resistances and non-linear current-voltage (*I-V*) curves^{27,28} as a result of insulating oxide layers or organic capping ligands between the NWs.

10 The formation of electrically-active connections between semiconductor NWs grown by a vapor-liquid-solid (VLS) mechanism has been limited to junctions formed either by electrical biasing individual wires,²⁹ patterning NWs to intersect during the VLS process,³⁰⁻³² or using a multi-step VLS processes to create branched nanowires³³⁻³⁵. However, these strategies are generally
15 limited to a low number of NWs and interconnection points.

What is needed are new and improved methods of joining separate semiconductor NWs on a large scale with crystalline and Ohmic junctions after VLS growth. Efficient and effective methods of synthesizing electrical connections between semiconductor nanowires NWs on a large scale to
20 form a network of interconnected NWs is needed. These and other unmet needs are provided by the instant disclosure.

SUMMARY

This summary includes several embodiments of the presently
25 disclosed subject matter, and in many cases lists variations and permutations of these embodiments. This summary is merely exemplary of the numerous and varied embodiments. Mention of one or more representative features of a given embodiment is likewise exemplary. Such an embodiment can typically exist with or without the feature(s) mentioned;
30 likewise, those features can be applied to other embodiments of the presently disclosed subject matter, whether listed in this summary or not. To avoid excessive repetition, this summary does not include or suggest all possible combinations of such features.

In some embodiments, provided herein are methods of synthesizing electrical connections between semiconductor nanowires (NWs) by capillarity-induced welding, the methods comprising creating or providing two or more NWs, aligning the two or more NWs to yield at least one inter-nanowire point-of-contact on each NW, and welding the two or more NWs together at the at least one inter-nanowire point-of-contact on each NW by capillarity-driven surface diffusion to form an electrical connection between the two or more NWs, wherein the electrical connection forms an Ohmic junction between the two or more NWs. In some embodiments the welding the two or more NWs together creates at least one inter-nanowire point-of-contact on each NW by capillarity-driven surface diffusion.

In some embodiments, provided herein are flexible and conductive thin NW films formed by the disclosed methods, wherein the NWs in the film have Ohmic junctions.

In some embodiments, provided herein are NW film materials, comprising a plurality of NWs overlapping in a random orientation to form a film material, wherein the NWs in the film material are welded at a point-of-contact where two or more NWs overlap to form an electrical connection therebetween, wherein the electrical connections between the plurality of overlapping NWs forms Ohmic junctions.

Embodiments of the presently disclosed subject matter having been stated hereinabove, and which is achieved in whole or in part by the presently disclosed subject matter, other embodiments will become evident as the description proceeds when taken in combination with the accompanying Examples as best described hereinbelow.

BRIEF DESCRIPTION OF THE DRAWINGS

The presently disclosed subject matter can be better understood by referring to the following example figures (also "FIGS."). The components in the figures are not necessarily to scale, emphasis instead being placed upon illustrating the principles of the presently disclosed subject matter (often schematically). In the figures, like reference numerals designate corresponding parts throughout the different views. A further understanding

of the presently disclosed subject matter can be obtained by reference to an embodiment set forth in the illustrations of the accompanying drawings. Although the illustrated embodiment is merely exemplary of systems for carrying out the presently disclosed subject matter, both the organization
5 and method of operation of the presently disclosed subject matter, in general, together with further objectives and advantages thereof, may be more easily understood by reference to the drawings and the following description. The drawings are not intended to limit the scope of this presently disclosed subject matter, which is set forth with particularity in the
10 claims as appended or as subsequently amended, but merely to clarify and exemplify the presently disclosed subject matter.

For a more complete understanding of the presently disclosed subject matter, reference is now made to the following drawings.

FIGS. 1A through 1C are schematic illustrations of a three-step
15 process, including NW growth, collapse, and welding, to create conductive semiconductor NW networks.

FIGS. 2A and 2B are transmission electron microscopy (TEM) images of two junctions (labeled by arrows) formed between three crossed NWs, with FIG 2A being a high-resolution transmission electron microscopy (HR-
20 TEM) image, and FIG. 2B being an energy-dispersive x-ray spectroscopy (EDS) elemental map of oxygen (O) using scanning TEM (STEM).

FIGS. 2C through 2H are electron microscopy images of NWs illustrating properties of a single, welded junction between two NWs. FIG 2C is a TEM image and FIGS. 2D, 2E and 2F are EDS elemental maps of Si, O, and P, respectively, for a single, welded NW junction within the network of
25 NW junctions. FIG. 2G is a combined Si and O map, and FIG. 2H is a corresponding elemental line scan.

FIGS. 3A, 3C and 3D are high-resolution TEM images of a crossed junction of two NWs, while FIG. 3B is a schematic of the image of FIG. 3A.

30 FIGS. 4A and 4B are TEM images of two adjacent, welded NWs, with FIG. 4B being a close-up view of a portion of FIG. 4A.

FIGS. 5A through 5K illustrate the diameter and temperature dependence of the welding process. FIG. 5A is a plot showing the suitable

temperature for welding dependent on the diameter of the wire to be welded, where circles represent optimal temperatures, squares represent temperatures too low, and triangles represent temperatures too high. FIGS. 5B through 5D are SEM images of optimally welded NWs of different diameters. FIGS. 5E through 5G are SEM images of NWs with the approximately same diameter welded at temperatures too high, optimal, and too low, respectively. FIG 5H is an SEM image of welded Ge NWs. FIGS. 5I through 5K are simulated schematics of the welding process as time increases, or as temperature increases for fixed time.

FIGS. 6A through 6F illustrate the electrical transport properties and recombination dynamics in single, welded Si junctions. FIGS. 6A and 6B are SEM images of electrodes on two NWs with a single welded junction. FIG 6C is a plot of the result of testing the electrical transport properties of a single junction on two welded NWs. FIGS. 6D through 6F are plots of the results of the testing the recombination dynamics of a single junction on two welded NWs.

FIGS. 7A and 7B are cross-sectional SEM images of collapsed and welded NWs about 30 nm (FIG. 7A) and about 60 nm (FIG. 7B) in diameter.

FIGS. 8A through 8C are schematic illustrations of the fabrication of flexible and conductive Si NW networks.

Figure 9 is a plot of the resistance multiplied by channel width as a function of electrode separation (*i.e.* channel length) for about 50 nm diameter NW networks measured on the Si growth substrate (squares) and on the substrate-free NW-PDMS film (triangles).

Figure 10 is a plot of the *I*-*V* data for the NW-PDMS film when straight and in a bent conformation with a radius of curvature of about 4 mm.

DETAILED DESCRIPTION

The presently disclosed subject matter now will be described more fully hereinafter, in which some, but not all embodiments of the presently disclosed subject matter are described. Indeed, the presently disclosed subject matter can be embodied in many different forms and should not be construed as limited to the embodiments set forth herein; rather, these

embodiments are provided so that this disclosure will satisfy applicable legal requirements.

The terminology used herein is for the purpose of describing particular embodiments only and is not intended to be limiting of the presently disclosed subject matter.

All technical and scientific terms used herein, unless otherwise defined below, are intended to have the same meaning as commonly understood by one of ordinary skill in the art. References to techniques employed herein are intended to refer to the techniques as commonly understood in the art, including variations on those techniques or substitutions of equivalent techniques that would be apparent to one of skill in the art. While the following terms are believed to be well understood by one of ordinary skill in the art, the following definitions are set forth to facilitate explanation of the presently disclosed subject matter.

In describing the presently disclosed subject matter, it will be understood that a number of techniques and steps are disclosed. Each of these has individual benefit and each can also be used in conjunction with one or more, or in some cases all, of the other disclosed techniques.

Accordingly, for the sake of clarity, this description will refrain from repeating every possible combination of the individual steps in an unnecessary fashion. Nevertheless, the specification and claims should be read with the understanding that such combinations are entirely within the scope of the invention and the claims.

Following long-standing patent law convention, the terms "a", "an", and "the" refer to "one or more" when used in this application, including the claims. Thus, for example, reference to "a nanowire" includes a plurality of such nanowires, and so forth.

Unless otherwise indicated, all numbers expressing quantities of ingredients, reaction conditions, and so forth used in the specification and claims are to be understood as being modified in all instances by the term "about". Accordingly, unless indicated to the contrary, the numerical parameters set forth in this specification and attached claims are approximations that can vary depending upon the desired properties sought

to be obtained by the presently disclosed subject matter.

As used herein, the term “about,” when referring to a value or to an amount of a composition, mass, weight, temperature, time, volume, concentration, percentage, *etc.*, is meant to encompass variations of in some
5 embodiments $\pm 20\%$, in some embodiments $\pm 10\%$, in some embodiments $\pm 5\%$, in some embodiments $\pm 1\%$, in some embodiments $\pm 0.5\%$, and in some embodiments $\pm 0.1\%$ from the specified amount, as such variations are appropriate to perform the disclosed methods or employ the disclosed compositions.

10 The term “comprising”, which is synonymous with “including” “containing” or “characterized by” is inclusive or open-ended and does not exclude additional, unrecited elements or method steps. “Comprising” is a term of art used in claim language which means that the named elements are essential, but other elements can be added and still form a construct
15 within the scope of the claim.

As used herein, the phrase “consisting of” excludes any element, step, or ingredient not specified in the claim. When the phrase “consists of” appears in a clause of the body of a claim, rather than immediately following the preamble, it limits only the element set forth in that clause; other
20 elements are not excluded from the claim as a whole.

As used herein, the phrase “consisting essentially of” limits the scope of a claim to the specified materials or steps, plus those that do not materially affect the basic and novel characteristic(s) of the claimed subject matter.

25 With respect to the terms “comprising”, “consisting of”, and “consisting essentially of”, where one of these three terms is used herein, the presently disclosed and claimed subject matter can include the use of either of the other two terms.

As used herein, the term “and/or” when used in the context of a listing
30 of entities, refers to the entities being present singly or in combination. Thus, for example, the phrase “A, B, C, and/or D” includes A, B, C, and D individually, but also includes any and all combinations and subcombinations of A, B, C, and D.

As used herein, nanowires (NWs) can in some embodiments refer to any high-aspect ratio structure with a cross-sectional size of about 1 micron or less and with variable cross sectional shapes, including for example a circle, ellipse, square, rectangle, hexagon, octagon, etc.

5 It will be understood that various details of the presently disclosed subject matter may be changed without departing from the scope of the presently disclosed subject matter. Furthermore, the foregoing description is for the purpose of illustration only, and not for the purpose of limitation.

10 The presently disclosed subject matter now will be described more fully hereinafter, in which some, but not all embodiments of the presently disclosed subject matter are described. Indeed, the presently disclosed subject matter can be embodied in many different forms and should not be construed as limited to the embodiments set forth herein; rather, these embodiments are provided so that this disclosure will satisfy applicable legal
15 requirements.

Provided herein are methods, systems and processes for synthesizing electrical connections between semiconductor NWs on a growth wafer, or other suitable substrate as discussed herein, by a capillarity-induced welding process, forming a complex network of interconnected NWs. More particularly,
20 demonstrated herein is a complementary new method to join separate semiconductor NWs on a large scale after vapor-liquid-solid (VLS) growth (or other mechanism for growing NWs) such that the resulting electrically connected NWs have crystalline and Ohmic junctions.

In some embodiments such methods of synthesizing electrical
25 connections between semiconductor NWs by capillarity-induced welding can comprise creating, i.e. synthesizing, or otherwise providing, two or more NWs, and aligning the two or more NWs to yield at least one inter-nanowire point-of-contact on each NW. Such an alignment can be random or intentional so long as the NWs overlap at least at one point to form a point-of-contact or junction
30 point. At the point-of-contact the two or more NWs can be welded together by capillarity-driven surface diffusion to form an electrical connection or junction between the two or more NWs. The electrical connection forms an Ohmic junction between the two or more NWs. Completing these steps on a large

scale for multiple NWs can allow for the synthesis of a network of interconnected NWs having Ohmic junctions.

In this process, as illustrated in FIGS. 1A through 1C, NWs can be grown by, for example, a VLS mechanism using Au catalysts and then collapsed using liquid capillary forces to yield multiple inter-nanowire points-of-contact on each NW. NWs can in some embodiments be welded by a capillarity-driven surface diffusion process for about 1 minute to about 240 minutes, or about 1, 2, 3, 4, 5, 10, 15, 20, 25, 30, 60, 120 or 240 minutes, at temperatures ranging from about 100°C to about 1,000°C below the bulk melting point, or about 100, 200, 300, 400, 500, 600, 700, 800, 900 or 1,000°C below the bulk melting point, in some embodiments about 4 minutes at temperatures of about 400-600°C below the bulk melting point (see Methods for all experimental details). NWs grown by other mechanisms and systems other than VLS are equally applicable to the disclosed methods of welding by capillarity-driven surface diffusion.

These methods and systems are illustrated in the schematic illustrations of FIGS. 1A through 1C. FIG. 1A illustrates that NWs **102** can be provided or grow on a substrate or growth substrate **104** using any suitable NW growth technique including those discussed herein. Once grown or synthesized, NWs **102** can be collapsed **C** using any suitable collapse method including but not limited to those described herein. See, e.g. FIG. 1B. During the collapse **C** procedure NWs **102** are randomly oriented along a planar surface of substrate **104**. Within this three-dimensional layer of randomly oriented NWs **102** the NWs **102** overlap one another forming points-of-contact **106**, or overlaps, as shown in FIG. 1B. By using a welding **W** technique or method as disclosed herein, a weld portion or junction **108** is formed at the overlap of each NW **102**. As shown in FIG. 1C, once welded **W** at each point-of-contact **106**, the three-dimensional layer of randomly oriented NWs **102** forms a three-dimensional film of joined NWs **102**, as discussed further herein.

Although the disclosed methods of forming a complex network of interconnected NWs is not intended to be limited by the method of forming the NWs initially, one example method of NW synthesis includes the vapor-liquid-solid (VLS) process. In some embodiments such a VLS process can comprise

a two-step growth procedure that avoids vapor-solid overcoating on a NW surface. In some embodiments VLS NW synthesis can include the creation of NWs on a growth wafer or other suitable substrate. The NWs can also be doped with phosphorus (P) to create doped or degenerately-doped n-type Si
5 NWs, or doped with boron (B) to create doped or degenerately-doped p-type Si. The NWs can be doped with any material typical used in the art. The NWs can be composed of a semiconductor material such as Si or Ge, or any other semiconductor material made in a NW morphology.

By way of example and not limitation, other suitable methods of
10 synthesizing and/or creating NWs for the disclosed capillarity-driven welding methods include a top-down fabrication method, a vapor-phase deposition process (such as but not limited to chemical vapor deposition, metal organic chemical vapor deposition, vapor-phase epitaxy, and molecular beam epitaxy), a liquid-phase deposition process, and an electrochemical
15 deposition process. Regardless of the method used, in some embodiments suitable NWs can comprise any high-aspect ratio structure with a cross-sectional size of about 1 micron or less and with variable cross sectional shapes, including a circle, ellipse, square, rectangle, hexagon and/or octagon.

20 For NWs provided by synthesizing them on a substrate, the alignment of the NWs prior to welding can in some aspects comprise a step of collapsing the NWs on the growth substrate. In some aspects collapsing the NWs comprises using a liquid capillary force, such as for example using liquid nitrogen or other inert or passivating liquid to cause the collapse of the
25 NWs on the growth substrate. In some applications the use of liquid nitrogen can be suitable, particularly since in some embodiments the use of liquid nitrogen can avoid or substantially avoid the formation of an oxide layer on the NW surface prior to welding. However, any procedure sufficient to cause the NWs to touch, overlap and/or otherwise come into sufficient contact with
30 a point of curvature sufficient for the subsequent welding can be used. Depending on the procedure used, and/or the concentration of the NWs, in some embodiments a plurality of inter-nanowire points-of-contact on each NW can be achieved.

As an alternative to synthesizing and/or collapsing grown NWs prior to the welding process, suitable NWs can also be provided in a suspended solution and then deposited on a substrate in a random or oriented manner causing the NWs to overlay or touch to form inter-nanowire points-of-contact
5 between the NWs. Any suitable method of arranging a plurality of NWs such that they are overlapping and/or contacting one another in a random or organized manner is within the scope of the instant disclosure and provides a suitable arrangement of NWs for the disclosed welding method.

As discussed herein, the NWs can in some embodiments be welded
10 by a capillarity-driven surface diffusion process over a range of times and temperatures depending on the desired outcome and operating conditions. As discussed in the Examples, in some embodiments the NWs are welded by capillarity-driven surface diffusion for about 1 minute to about 10 minutes at a temperature of about 400°C to about 600°C below a bulk melting point.
15 Such welding reshapes the inter-nanowire point-of-contact between each NW to form a high density of interconnections. The welding reshapes the inter-nanowire point-of-contact between each NW to form a compositionally-uniform and oxygen-free interface between each NW. Notably, the interface formation caused by the welding is a self-limited process (see Examples for
20 further details). In some aspects, the disclosed methods can further comprise removing any oxide layer present on the NWs prior to welding.

The disclosed methods and systems for capillarity-driven welding of semiconductor nanowires for flexible three-dimensional networks is based at least in part on the discovery that such methodologies result in NW networks
25 with Ohmic junctions. As disclosed in more detail in the Examples, such NW networks or films can have an *I-V* curve that is substantially linear over a broad voltage range. Moreover, the volume density of Ohmic junctions can meet or exceed $1\ \mu\text{m}^{-3}$. Such conductive films are three dimensional and can have a thickness of about 5 μm to about 10 μm , or in some
30 embodiments ranging from about the thickness of one NW to 100 μm or more.

As shown in FIGS. 8A through 8C, and discussed in the Examples, fabrication of flexible and conductive Si NW networks can in some

embodiments comprise a NW-PDMS lift-off process. Such a process can comprise, for example, PDMS deposition on the growth wafer (FIG. 8A), wet-chemical etching of Ge (FIG. 8B), and lift off of the NW-PDMS film (FIG. 8C). Large-area, flexible networks can be created by depositing a sacrificial
5 germanium (Ge) layer **308** on a growth wafer **306** prior to Au catalyst dispersal. This layer can be removed by wet-chemical etching **304** in hydrogen peroxide (FIG. 8B), releasing the network **302** from the substrate **306** to create a free-standing film **310** (FIG. 8C). In some embodiments, to facilitate handling of the lightweight NW networks/films, the NW can be
10 infiltrated with poly-dimethyl siloxane (PDMS) prior to Ge etching to provide additional handling stability, leading to the lift-off process illustrated in FIGS. 8A through 8C. The resulting NW film can in some embodiments have high optical transparency, which matches finite-element optical simulations of the transmittance spectrum. The transparency and color of the NW films can in
15 some embodiments strongly depend on diameter-dependent Lorentz-Mie scattering, and optical tuning is possible simply by changing the diameters in the sample.

In some embodiments disclosed herein Si and Ge NWs are used as examples. However, the presently disclosed methods, systems, processes
20 and resulting products are not limited to Si and Ge NWs, but are applicable to any element or material suitable for NW synthesis under the conditions disclosed and discussed herein. For example, in addition to Si and Ge NWs, suitable NWs can be made of semiconductors including but not limited to group IV, group III-V, group II-VI, and group IV-VI semiconductors as well
25 as alloys and combinations thereof. In some embodiments these may be Si, Ge, $\text{Si}_x\text{Ge}_{1-x}$, GaN, GaP, GaAs, GaSb, AlN, AlP, AlAs, AlSb, InN, InP, InAs, InSb, ZnO, ZnS, ZnSe, CdO, CdS, CdTe, PbO, PbS, PbSe, and alloys thereof.

While liquid nitrogen can in some embodiments be used in the collapse
30 process, this is only one exemplary procedure. In some embodiments any collapse procedure can be used so long as the synthesized NWs are caused to touch, overlap or otherwise come into sufficient contact with a point of curvature sufficient for the subsequent welding procedure. By way of example

and not limitation, NWs can be suspended in a solution and then deposited on a substrate in a random or oriented manner causing them to overlay or touch.

Thus, demonstrated herein is a capillarity-driven welding of semiconductor NWs, and the networking of such NWs on a large scale to
5 create flexible and conductive thin films. Because of the capillarity-driven surface-diffusion mechanism, this welding process is generic for all types of semiconductor NWs (Si and Ge NWs used in the examples herein are exemplary only and not limiting).

Such NW networks can be used in solar energy and battery electrodes,
10 bioelectronic scaffolds, and a platform for neuromorphic computation. For instance, the volume density of junctions can meet or exceed $1 \text{ } \mu\text{m}^{-3}$, which is comparable to the density, about 10^9 mm^{-3} , of synapses in the human brain⁵⁵. Although inherent disorder is often seen as an impediment to implementing semiconductor nanomaterials in realistic device applications, semiconductor
15 NW networks have promise for a variety of applications without the use of advanced lithography or assembly methods.

EXAMPLES

The following examples are included to further illustrate various
20 embodiments of the presently disclosed subject matter. However, those of ordinary skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments which are disclosed and still obtain a like or similar result without departing from the spirit and scope of the presently disclosed subject matter.

25

Materials and Methods

Synthesis of NWs and networks.

A home-built chemical vapor deposition (CVD) system was used for all syntheses and includes a quartz-tube furnace (Lindberg Blue M),
30 hydrogen (H_2) and Argon (Ar) carrier gases (Matheson Trigas; 5N semiconductor grade), and reactive gases (Voltaix/Air Liquide Advanced Materials) silane (SiH_4), germane (GeH_4 ; 10% in H_2), diborane (B_2H_6 ; 100 ppm in H_2), and phosphine (PH_3 ; 1000 ppm in H_2). NW growth substrates

consisted of Si/SiO₂ wafers (Nova Electronic Materials; B-doped 1-10 Ω·cm Si wafers with 600 nm thermal oxide) and were optionally coated with about 200 nm sacrificial Ge layer following a modified literature procedure⁵⁰. Briefly, substrates were annealed for 10 minutes at 600°C with 100 standard
5 cubic centimeters per minute (sccm) of H₂, a seed layer was deposited for 1 min at 350°C and 20 Torr using 1 sccm SiH₄ and 10 sccm B₂H₆ with 60 sccm Ar, and Ge was deposited for 75 min at 330°C and 5 Torr using 10 sccm of GeH₄ and 100 sccm H₂. The Si/SiO₂ or Si/SiO₂/Ge substrates were then placed in an ultraviolet/ozone cleaner (Samco UV-1) and functionalized
10 with a 10:1 water: poly-L-lysine (0.1% in H₂O, Sigma Aldrich) solution followed by dispersion of citrate-stabilized Au colloids (BBI international) with diameters of about 30-100 nm. N-type Si NWs were grown in the CVD system following a modified literature procedure^{21,27} using a 5 min 450°C nucleation step followed by a ramp (1 °C/min cooling rate) to 420°C with 200
15 sccm H₂, 2 sccm SiH₄, 10 sccm PH₃, and a total growth time of 5 hours.

NW arrays were collapsed by sliding the growth substrate directly from the quartz tube into liquid nitrogen (N₂) while flowing 200 sccm Ar. The substrate was then reinserted into the quartz tube, and the tube was evacuated to vacuum. Evaporation of liquid N₂ caused collapse of the NWs
20 without formation of an oxide layer.

The welding process was performed by raising the temperature to 800-900°C under 200 sccm H₂ at 8 Torr. At the desired temperature, the H₂ flow was reduced to 60 sccm, the pressure was ramped to 25 Torr, the conditions were held for 4 min, and the system was then cooled to room
25 temperature. Ge NW networks were synthesized by modifying the procedure for Si NWs. Ge NWs were nucleated for 30 min at 320°C under 100 sccm Ar and 15 sccm GeH₄ at 300 Torr followed by a ramp (5°C/min cooling rate) to 260°C and continued growth for 4 hours. Ge networks were welded using the same procedure as for Si NWs but with a temperature of 600°C rather than
30 800-900°C.

Electron microscopy imaging.

SEM imaging was performed using an FEI Helios 600 Nanolab Dual Beam system. Samples for HR-TEM and STEM were prepared by mechanical contact-transfer directly on to lacey-carbon TEM grids (Ted-Pella #01895). STEM imaging was performed on a Tecnai Osiris operating at 200 kV with a sub-nm probe with a current of 2 nA (spot size 3, 4k extraction voltage). Drift-corrected STEM-EDS maps were obtained using the Bruker Esprit software. Acquisition time for each map was about 15 minutes. Standardless Cliff-Lorimer quantification was performed on the deconvoluted spectra from sub-sections of the EDS maps. STEM images were obtained before and after map acquisition to note any change in the sample.

Simulations.

The geometric models were created using version 2.70 of the program Surface Evolver⁴¹. The initial crossed NW geometry begins as a simplicial complex constructed from two perpendicular icosahedral prisms that intersect at four points to form a continuous body. The surface is refined and allowed to evolve towards a minimal energy configuration via a gradient descent method under isochoric conditions. The three simulations results in FIGS. 5I through 5K represent successive time points along a progression dictated by the mean curvature. The models were imported into COMSOL Multiphysics, and the surface gradient of the curvature was calculated with a weak-form boundary partial differential equation formulation. The surface velocity, v_s , was then calculated as given by equation 2 (below).

Flexible network fabrication.

For fabrication of NW-PDMS films, Si/SiO₂/Ge substrates were used for NW growth, and PDMS (Sylgard 184 Silicone Elastomer) was spun cast onto the NW networks (500 rpm for 5 seconds and 1500 rpm for 30 seconds). The substrates were cured for 60 min at 60°C. Polymer was removed from the edges of the substrate, and the substrate was immersed in 30% hydrogen peroxide at 80°C for several days to remove the Ge layer.

Transport measurements.

For measurements on NW networks, titanium/palladium (Ti/Pd) contacts (5/200 nm) were evaporated, after a brief BHF etch, onto the exposed NW networks via a shadow mask in an electron beam evaporator

(Thermionics VE-100). For single-junction measurements, welded NWs were mechanically transferred onto device substrates (Nova Electronic Materials; B-doped 1-10 Ω -cm Si wafers with 100 nm thermal oxide and 200 nm silicon nitride). Electron-beam lithography was used to pattern electrical contacts to individual NWs, and Ti/Pd contacts (3/200 nm) were evaporated after a brief etch in BHF. *I-V* data for both NW networks and single welded junctions were collected with a Keithley 2636A SourceMeter in conjunction with Signatone micropositioners (S-725) and probe tips (SE-TL) or a Lake Shore Cryotronics PS-100 probe station. NW diameters and channel lengths were measured by SEM. For measurements in a bent configuration, NW-PDMS films were wrapped around a pencil to demonstrate the durability of the networks.

Ultrafast microscopy.

Welded intrinsic Si NW junctions were mechanically transferred onto glass slides. A mode-locked Ti:Sapphire oscillator (80 MHz) generated an 850 nm output, which was split by a 10/90 beam splitter. The high-power beam was frequency doubled to 425 nm and served as the pump, while the lower power beam was used as the probe. Repetition rates of pump and probe beams were reduced to 1.6 MHz using two synchronized acousto-optic modulator (AOM) pulse pickers. The pump beam was modulated at 16 kHz using the AOM. The 10 pJ/pulse pump beam and 1.5 pJ/pulse probe beam were recombined using a dichroic beam splitter and directed onto the back aperture of a 50x (0.8 NA) objective that focused them to diffraction-limited spots on the welded structures. The probe beam was collected with a condenser lens, filtered to remove pump light, and directed onto a balanced photodiode. Pump-induced changes in the intensity of the probe pulse were monitored with a digital lock-in amplifier. For kinetic traces, the probe beam was increased in intensity to 2.5 pJ/pulse. Errors for the reported time constants reflect two standard deviations.

Absorbance data.

Junction transmittance spectra were measured with a microspectrophotometer (20/30 PV UV-Visible-NIR from Craic Industries) using a 10x objective (Ultrafluor 10x 0.2 NA 7.4 mm WD) in conjunction with

a xenon arc lamp source and CCD array detector. Optical simulations were performed using COMSOL Multiphysics. The two-dimensional simulation domain consisted of a Si NW with a circular cross-section placed in a vacuum encapsulated by perfectly matched layers (PMLs) to prevent reflection/scattering effects from the simulation boundaries. Absorption characteristics of single NWs were simulated by impinging a plane wave with either transverse electric (TE) or transverse-magnetic (TM) polarization using the total-field, scattered-field method. A polarization-independent transmittance was calculated by averaging the TE and TM transmittance.

The simulation was repeated for NW diameters from 20-145 nm in steps of 3 nm.

Example 1

Methodology for capillarity-driven welding of semiconductor nanowires for flexible three-dimensional networks with ohmic interconnects

In this process, as illustrated in FIGS. 1A through 1C, a three-step process, including NW growth, collapse, and welding, can create conductive semiconductor NW networks. In some embodiments NWs can first be grown by the VLS mechanism using Au catalysts and then collapsed using liquid capillary forces to yield multiple inter-nanowire points-of-contact on each NW, or substantially each NW. Then, in some embodiments, the NWs can be welded by a capillarity-driven surface diffusion process for about 4 minutes at temperatures of about 400-600 °C below the bulk melting point (see Methods for all experimental details).

In the schematic illustrations of FIGS. 1A through 1C, NWs **102** can be provided or grow on a substrate or growth substrate **104** using any suitable NW growth technique including those discussed herein. Once grown or synthesized NWs **102** can be collapsed **C** using any suitable collapse method including but not limited to those described herein. During the collapse **C** procedure NWs **102** are randomly oriented along a planar surface of substrate **104**. Within this three-dimensional layer of randomly oriented NWs **102** the NWs **102** overlap one another forming points-of-contact **106**, or overlaps, as shown in FIG. 1B. By using a welding **W** technique or method as disclosed herein a weld portion or junction **108** is formed at the overlap of each NW **102**.

Once welded **W** at each point-of-contact **106**, the three-dimensional layer of randomly oriented NWs **102** forms a three-dimensional film of joined NWs **102**, as discussed further herein.

For the Si and Ge NWs used in this study, the VLS process was performed using a two-step growth procedure that avoids vapor-solid overcoating on the wire surface²². In some embodiments NWs were doped with phosphorus (P) to create degenerately-doped n-type Si. The collapse process was performed with liquid nitrogen to avoid formation of an oxide layer on the wire surface prior to welding. After completion of the welding process, the NW networks were exposed to ambient conditions, causing formation of a 2-3 nm native oxide on the surface.

Example 2

Microscopic imaging and analysis of welded NWs

FIGS. 2A and 2B show a high-resolution transmission electron microscopy (HR-TEM) image (FIG. 2A) and an energy-dispersive x-ray spectroscopy (EDS) elemental map (FIG. 2B) of oxygen (O) using scanning TEM (STEM) of two junctions (labeled by arrows) formed between three crossed NWs. The welding process reshapes the points-of-contact between NWs, and a clear junction is observed, forming a high density of interconnections.

FIGS. 2C through 2H further illustrate properties of a single, welded junction between two NWs. Particularly, FIG 2C is a TEM image and FIGS. 2D, 2E and 2F are EDS elemental maps of Si, O, and P, respectively, for a single, welded NW junction within the network of NW junctions. These images highlight several features. First, the TEM image (FIG. 2C) shows an increase in the NW volume in the vicinity of the junction, and the Si map (FIG. 2D) shows that the junction region contains approximately twice the Si content of the intersecting NWs. Second, the P map (FIG. 2E) shows that the P dopants are uniformly distributed throughout the wire and junction without any segregation at surfaces or interfaces. Third, the O map (FIG. 2F) shows that a native oxide layer has formed uniformly on the surface of the wires and the junction but not at the interface between the two wires, as

evidenced by the O signal at the junction being equivalent in amplitude to the signal along the individual NWs. A combined Si and O map (FIG. 2G), and corresponding elemental line scan (FIG. 2H), of a second welded junction further confirm the absence of an oxide layer at the interface
5 between the wires.

Example 3

Compositional analysis of welded NW junctions

The images in FIGS. 2C through 2H demonstrate that the two wires
10 have locally fused to form a compositionally-uniform and oxygen-free interface. High-resolution TEM images of a second crossed junction (FIG. 3A) of two NWs, NW **102a** and NW **102b**, corroborate this observation. Lattice-resolved images indicate that each NW, NW **102a** and NW **102b**, shown in FIG. 3A was grown in the [112] direction, and junction **108** consists
15 of the two crystal structures overlaid (depicted schematically in FIG. 3B) without any polycrystalline or amorphous material. FIGS. 3C and 3D are close-up views of insets 1 and 2 from FIG. 3A showing junction **108** of NW **102a** and NW **102b**.

Additional TEM images for two adjacent, welded NWs are shown in
20 FIGS. 4A and 4B. The image of FIG 4A shows that the process can in some embodiments fuse two NWs, NW **102c** and **102d**, leaving a single zig-zag grain boundary or junction. FIG. 4B is a close-up view of inset **124** in FIG. 4A.

Example 4

NW diameter and temperature dependence analysis

The diameter and temperature dependence of the welding process was examined, as shown in FIGS. 5A through 5K. The temperature suitable in some embodiments for welding shows a strong dependence on diameter, and the qualitative results from multiple wire diameters and temperatures are
30 summarized in FIG. 5A. Three regimes were observed: absence of welding (squares below dashed line), optimal welding (circles above dashed line but below dashed-dotted line), and structural instability (triangles above dashed-dotted line). As shown by the SEM images in FIGS. 5B, 5C and 5D, NWs

with diameters of about 100, about 50, and about 30 nm welded at temperatures of about 875, about 840, and about 840°C, respectively. For each diameter, temperatures of about 25°C higher resulted in structural instability at the junction (FIG. 5E) whereas temperatures about 25°C lower
5 resulted in no noticeable morphological change (FIG. 5G), as exemplified by the SEM images in FIGS. 5E through 5G for NWs of about 80 nm in diameter.

The morphology of the weld as a function of the process time was also examined. Although 4 minutes was typically used, longer weld times
10 resulted in no additional change to the junction or NW morphology for the optimal weld conditions, indicating that the junction formation can in some embodiments be a self-limited process. In addition, no welding was observed for NWs that had been exposed to ambient conditions prior to the weld process, an observation that in some embodiments can be attributed to
15 formation of a thin native oxide layer that inhibits the welding mechanism. The welding process was also performed on Ge nanowires (FIG. 5H), and a substantially lower temperature of 600°C was necessary to create high-quality junctions. The results on Si and Ge NWs indicates that the weld process can in some embodiments be successful with high-quality, oxide-
20 free NW surfaces at temperatures of about 400-600°C below the bulk melting temperature of the NW material.

Without being bound by any particular theory or mechanism of action, in some embodiments the strong dependence of the weld temperature on diameter suggests that the weld results from a physical process that
25 depends on surface curvature. Capillarity-induced surface diffusion, a process in which a change in curvature creates a free-energy gradient that drives atoms away from areas of high curvature, can in some embodiments occur in spherical and cylindrical nanostructures. The effects of this phenomenon can in some embodiments include the blunting of W tips and
30 necking or sintering of nanoparticles. To understand the possible role of this phenomenon for the welding of NWs, the diffusion process for realistic NW geometries was modeled. The surface diffusion flux, J_s , is related to the local curvature, K , by:

$$J_s = - (D_s(T)\gamma\Omega^{2/3}/k_B T)\nabla_s K ,$$

(Equation 1)

5 where K is $(1/R_1+1/R_2)$, R_1 and R_2 are the principle radii of curvature for a two-dimensional surface, $D_s(T)$ is the temperature-dependent surface diffusion coefficient, γ is the surface tension (1.6 N/m), Ω is the atomic volume of .020 nm³, k_B is the Boltzmann constant, T is temperature, and $\nabla_s K$ is the surface gradient of the curvature. The normal velocity of the surface,
 10 v_s , which describes the deformation of the NW junction, can be calculated as:

$$v_s = \Omega^{2/3} J_s ,$$

(Equation 2).

Simulations, as depicted in FIGS. 5I, 5J and 5K, were performed
 15 using a surface-evolving simulation⁴¹ combined with finite-element calculations of v_s (see Methods). Simulations of the welding process showing initial (FIG. 5I), intermediate (FIG. 5J), and final (FIG. 5K) junction formation. The axis reflects the velocity of the surface normal with positive (+10) and negative (-10) values indicating increasing and decreasing diameters,
 20 respectively; scale bars, 100 nm. Insets: cross-sectional profile of each simulation in the plane that longitudinally bisects the upper NW and passes through the center of the junction; scale bars, 50 nm.

As shown in the first simulation image (FIG. 5I) at a T of 850°C and corresponding D of 1.8×10^{-12} m²/s⁴², a large surface diffusional flux toward
 25 the junction area **108** of NWs **102a** and **102b** is predicted, producing a v_s as high as about 112 nm/s. Without being bound by any particular theory or mechanism of action, the process drives the system to reduce curvature by forming the welded geometry, as shown in the second (FIG. 5J) and third (FIG. 5K) simulation snapshots with maximum v_s of about 10 and about 3
 30 nm/s, respectively. Thus, in some embodiments the weld eliminates the high curvature interface between the two wires, such as in FIG. 5K, and the dramatic reduction of v_s as the weld evolves explains the self-limited nature of the process. In addition, the higher curvature in smaller-diameter crossed

junctions, causing higher v_s , can give rise to the diameter-dependence of the process. The presence of three regimes (no welding, optimal welding, and structural instability; FIG. 5A) over a relatively narrow temperature range can in some embodiments be explained by the Arrhenius dependence of $D_s(T)$, which for Si varies by one order of magnitude from 800 to 900°C. The welded junctions are consistent with the local sintering of Ag nNWs observed upon thermal annealing, and the structural instability is consistent with the onset of Plateau-Rayleigh instability and spheroidization of Ag NWs upon extended annealing. Furthermore, the capillarity-driven formation mechanism explains the absence of any morphological change in regions far from the junction that have nearly uniform curvature, for which v_s is approximately zero because there is negligible change in curvature (*i.e.* $\nabla_s K$ is zero).

Example 5

Analysis of electrical transport properties of welded NW junctions

FIGS. 6A through 6F illustrate the electrical transport properties and recombination dynamics in single, welded Si junctions. FIG. 6A is a SEM image of electrodes **E**, labeled 1-4, on two NWs, **102a** and **102b**, with a single welded junction **108**; scale bar, 2 μm . FIG. 6B is a SEM image of the junction **108** and the two electrodes **E** adjacent to the junction **108**; scale bar, 1 μm . FIG. 6C is a four-point probe I - V measurement of the junction shown in FIG. 6A collected by sourcing current on electrodes 1 and 4 and measuring the voltage across electrodes 2 and 3. FIG. 6D and 6E are SEM images of the NW junction probed by time-resolved pump-probe microscopy, showing the junction region (FIG. 6D; scale bar, 200 nm) and the entire region probed region (FIG. 6E; scale bar, 500 nm). Circles in FIG. 6E denote the regions in which kinetic traces were collected. Pump-probe images of charge carrier dynamics were collected at time delays of 0 ps, 13 ps, 27 ps and 67 ps, with all normalized relative to the maximum intensity at 0 ps, and analyzed, the results of which are shown in FIG. 6F. FIG. 6F shows the kinetic traces for pump-probe dynamics collected at regions denoted by the numbered circles in panel FIG 6C. The number 5 trace reflects kinetics at

the junction while other traces (1, 2, 3 and 4) represent dynamics in the four arms adjacent to the junction.

The electrical transport properties of a single junction were probed by fabricating electrodes (FIG. 6A and 6B) on two crossed NWs degenerately-doped at an encoded P doping level of $2.5 \times 10^{20} \text{ cm}^{-3}$. The I - V curve (FIG. 6C) collected using a four-point-probe configuration is linear and yields a junction resistance of $65 \pm 10 \text{ k}\Omega$. Table 1 provides details of the resistance measurements.

Table 1 | Variables for the calculation of the junction resistance

Junction	R_j (k Ω)	R_{exp} (k Ω)	ρ ($\Omega\text{-cm}$)	l_2 (μm)	l_3 (μm)	$r_{1,2}$ (nm)	$r_{3,4}$ (nm)
Figure 4a-b	69 ± 4	94.5 ± 0.2	$(1.8 \pm 0.3) \times 10^{-3}$	5.35 ± 0.03	4.69 ± 0.03	54 ± 2.5	43 ± 2.5

The junction resistance, R_j , was calculated from:

$$R_j = R_{exp} - \rho \left(\frac{l_2}{\pi r_{1,2}^2} + \frac{l_3}{\pi r_{3,4}^2} \right),$$

(Equation 3)

where ρ is the resistivity for a single NW segment doped at an encoded doping level of $5 \times 10^{20} \text{ cm}^{-3}$, R_{exp} is the measured resistance between contacts 2 and 3 as measured by the four-point probe configuration in Figure 4, l_2 is the distance from contact 2 to the junction, l_3 is the distance from contact 3 to the junction, $r_{1,2}$ is the radius of the NW with contacts 1 and 2, and $r_{3,4}$ is the radius of the NW with contacts 3 and 4. Radius and segment lengths were determined from SEM images. The resistivity ρ was determined from a four-point probe measurement (not shown) on a single NW taken from the same growth substrate as the welded NW junction. The resistivity value is in good agreement with prior measurements of the resistivity for NWs encoded at the same doping level,⁴⁴ and the value is consistent with an

active doping level of about $4 \times 10^{19} \text{ cm}^{-3}$. Table 1 summarizes all of the measured values for the NW device shown in FIGS. 6A and 6B.

This junction resistance demonstrates that the welding process forms relatively low-resistance and Ohmic connections between NWs. The resistance of the junction is approximately equal to the resistance associated with an about 30 μm length of a NW 100 nm in diameter, using the measured NW resistivity of about $0.0018 \text{ } \Omega\cdot\text{cm}$. The ratio of junction resistance to wire resistance is comparable to the ratio observed in Ag NWs, and for lower Si doping levels and thus higher resistivity wires, the junction resistance is expected to be negligible compared to the NW resistance.

The material quality in the vicinity of a welded junction (FIG. 6D) was probed using ultrafast pump-probe optical microscopy with a time resolution of about 500 fs, an experiment that has been shown to directly probe interfacial charge carrier (electron and hole) recombination dynamics in single NWs. In this experiment, a 425 nm pump pulse focused to a diffraction-limited spot photoexcites the NW at a specific spatial location, generating charge carriers that are probed by a time-delayed, spatially-overlapped probe pulse at 850 nm. The individual optical images were generated by raster scanning the spatially-overlapped pump-probe pulses over the NW junction at a fixed pump-probe time delay and measuring the change in intensity, ΔI , of the probe beam. The charge carriers induce an optical bleach ($\Delta I > 0$) that decays in amplitude as the photogenerated carriers recombine on a time scale of tens to hundreds of picoseconds. Kinetic traces collected at the center and four arms of the crossed junction (locations indicated by numbered circles) are displayed in FIG. 6F. The traces in the four arms are similar, giving rise to an average recombination time constant of $66 \pm 1 \text{ ps}$ when fit to single exponential functions. The kinetic trace in the junction produces a recombination time constant of $47 \pm 1 \text{ ps}$, which is about 29% shorter than the arms. The small difference between the recombination time in the junction and arms indicates a marginal decrease in the material quality, which is consistent with a grain boundary between the two NWs that slightly increases charge-carrier recombination. The combination of ultrafast microscopy data and electrical transport

measurements confirm the high electrical and material quality of the welded junctions.

Example 6

5 Synthesis of three-dimensional NW films

In some embodiments the network formation was scaled to the size of several square centimeters using substrates with millions of NW catalysts deposited randomly on the surface. FIGS. 7A and 7B are cross-sectional SEM images of collapsed and welded NWs about 30 nm (FIG. 7A) and
10 about 60 nm (FIG. 7B) in diameter. The Si substrates contain a 200 nm layer of Ge and 600 nm layer of SiO₂ (scale bars, 5 μm). The networks **202** formed are three-dimensional (FIG. 7A and 7B) with the effective thickness of the network **202** determined in some embodiments by the density or diameter, or combination of both, of the NWs. For high densities (>1 Au
15 catalyst particle per μm²), the diameter of the NW can in some embodiments strongly influence the thickness of the 3D network **202**, creating films of about 5 and about 10 μm thick for NW diameters of 30 and 60 nm, respectively, as exemplified by the SEM images in FIG. 7A and 7B.

FIGS. 8A through 8C are schematic illustrations of the fabrication of
20 flexible and conductive Si NW networks. More particularly, these figures are schematic illustrations of the NW-PDMS lift-off process, showing PDMS deposition on the growth wafer (FIG. 8A), wet-chemical etching of Ge (FIG. 8B), and lift off of the NW-PDMS film (FIG. 8C). Large-area, flexible networks were created by depositing an about 200 nm thick sacrificial germanium (Ge) layer **308** on a growth wafer⁵⁰ **306** prior to Au catalyst
25 dispersal. This layer can be removed by wet-chemical etching **304** in hydrogen peroxide, releasing the network **302** from the substrate **306** to create a free-standing film **310**. However, due to difficulties in handling the lightweight networks, they were infiltrated with poly-dimethyl siloxane
30 (PDMS) prior to Ge etching to provide additional handling stability, leading to the lift-off process illustrated in FIGS. 8A through 8C. The Ge layer **308** can be etched to release a flexible, free-standing NW-PDMS film **310**. The film can in some embodiments have high optical transparency, which matches

finite-element optical simulations of the transmittance spectrum. The transparency and color of the NW films can in some embodiments strongly depend on diameter-dependent Lorentz-Mie scattering, and optical tuning is possible simply by changing the diameters in the sample.

5 The electrical transport characteristics of the centimeter-scale NW networks or film **310** were probed before and after lift-off from the growth substrate. Networks were constructed of 50 nm diameter n-type NWs with an encoded doping level of $2.5 \times 10^{20} \text{ cm}^{-3}$, and they were grown on both Si/SiO₂ substrates and, for lift off, Si/SiO₂/Ge substrates.

10 Macroscopic electrodes were fabricated on both films to allow for testing. Figure 9 is a plot of the resistance multiplied by channel width as a function of electrode separation (*i.e.* channel length) for about 50 nm diameter NW networks measured on the Si growth substrate (squares) and on the substrate-free NW-PDMS film (triangles). Dashed lines represent
15 linear fits to the data (dashed corresponds to the squares, dashed-dotted corresponds to the triangles), and the vertical offset of the dashed line reflects a contact resistance of $30 \pm 13 \text{ k}\Omega\cdot\text{cm}$ for the NW-PDMS film. Inset: *I-V* curve measured on the Si growth substrate for an electrode separation of 1.5 mm. The substrate-free NW-PDMS film showed only a slight increase in
20 resistance compared to networks on the substrate.

 The approximate sheet resistance values of the two networks, as determined by the slope of the dashed lines in FIG. 9, are 170 ± 11 and $164 \pm 29 \text{ k}\Omega/\text{square}$ for the on-substrate (square) and substrate-free (triangles) films, respectively, showing no significant difference as a result of the lift-off
25 process. Thus, within the error of the measurements, the sheet resistances of the two films are the same. In addition, the fits yielded contact resistances from the y-intercepts of $2.2 \pm 4.4 \text{ k}\Omega\cdot\text{cm}$ and $30 \pm 13 \text{ k}\Omega\cdot\text{cm}$ for the on-substrate and substrate-free films, respectively. Considering the film widths of $15.2 \pm 0.4 \text{ mm}$ and $7.6 \pm 0.6 \text{ mm}$, the measurements yield contact
30 resistances of $1.4 \pm 2.9 \text{ k}\Omega$ and $39 \pm 17 \text{ k}\Omega$ for the on-substrate and substrate-free films, respectively. The contact resistance is thus zero within error for the on-substrate film but considerable for the substrate-free NW-PDMS film. Moreover, the *I-V* curves of the films are linear over a broad

voltage range (inset), demonstrating the high-quality of the Ohmic connections between the NWs.

Figure 10 is a plot of the I - V data for the NW-PDMS film when straight and in a bent conformation with a radius of curvature of about 4 mm. The performance of NW-PDMS films under strain was probed by bending the films. I - V data show a small, less than 50% increase in resistance under strain when measured across a 6 mm channel, demonstrating the mechanical robustness of the electrical conductivity through the network. This behavior is consistent with flexibility observed in Ag NW networks.

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All references listed herein including but not limited to all patents, patent applications and publications thereof, scientific journal articles, and database entries are incorporated herein by reference in their entireties to the extent that they supplement, explain, provide a background for, or teach methodology, techniques, and/or compositions employed herein.

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15 It will be understood that various details of the presently disclosed subject matter may be changed without departing from the scope of the presently disclosed subject matter. Furthermore, the foregoing description is for the purpose of illustration only, and not for the purpose of limitation.

CLAIMS

What is claimed is:

1. A method of synthesizing electrical connections between
5 semiconductor nanowires by capillarity-induced welding, the method comprising:
creating or providing two or more nanowires;
aligning the two or more nanowires to yield at least one inter-nanowire
point-of-contact on each nanowire; and
10 welding the two or more nanowires together at the at least one inter-nanowire point-of-contact on each nanowire by capillarity-driven surface diffusion to form an electrical connection between the two or more nanowires,
wherein the electrical connection forms an Ohmic junction between
15 the two or more nanowires.
2. The method of claim 1, wherein aligning the two or more nanowires comprises collapsing two or more nanowires on a growth substrate.
20
3. The method of claim 2, wherein collapsing the two or more nanowires comprises using a liquid capillary force, wherein the liquid capillary force comprises using liquid nitrogen or other inert or passivating liquid to cause the collapse of the two or more nanowires on the growth
25 substrate.
4. The method of claim 3, wherein collapsing the two or more nanowires comprises using liquid nitrogen, wherein using liquid nitrogen avoids formation of an oxide layer on the nanowire surface prior to welding.
30
5. The method of claim 3, wherein collapsing the two or more nanowires using the liquid capillary force comprises using a procedure sufficient to cause the two or more nanowires to touch, overlap and/or

otherwise come into sufficient contact with a point of curvature sufficient for subsequent welding.

6. The method of claim 1, wherein aligning the two or more
5 nanowires yields a plurality of inter-nanowire points-of-contact on each nanowire.

7. The method of claim 1, wherein electrical connections are
formed between a plurality of nanowires to form a network of interconnected
10 nanowires.

8. The method of claim 7, wherein a plurality of separate
nanowires are joined on a large scale with crystalline and/or Ohmic
junctions.
15

9. The method of claim 1, wherein the nanowires are created by a
vapor-liquid-solid process.

10. The method of claim 9, wherein the vapor-liquid-solid process
20 comprises a two-step growth procedure that avoids vapor-solid overcoating on a nanowire surface.

11. The method of claim 9, wherein the nanowires are created on a
growth wafer.
25

12. The method of claim 9, wherein the nanowires are doped with
phosphorus to create degenerately-doped n-type Si nanowires.

13. The method of claim 1, wherein the nanowires are welded by
30 capillarity-driven surface diffusion for about 1 minute to about 10 minutes at a temperature of about 400 °C to about 600 °C below a bulk melting point.

14. The method of claim 1, wherein the welding reshapes the inter-nanowire point-of-contact between each nanowire to form a high density of interconnections.

5 15. The method of claim 1, wherein the welding reshapes the inter-nanowire point-of-contact between each nanowire to form a compositionally-uniform and oxygen-free interface between each nanowire.

10 16. The method of claim 15, wherein the compositionally-uniform and oxygen-free interface caused by the welding is a self-limited process.

15 17. The method of claim 1, wherein the nanowires are made of materials selected from the group consisting of group IV, group III-V semiconductor materials, group II-VI semiconductor materials, group IV-VI semiconductor materials, and combinations or alloys thereof.

20 18. The method of claim 1, wherein the creating or providing two or more nanowires comprises providing the nanowires in a suspended solution and then depositing the nanowires on a substrate in a random or oriented manner causing the nanowires to overlay or touch to form at least one inter-nanowire point-of-contact on each nanowire.

25 19. The method of claim 1, comprising removing any oxide layer present on the nanowires prior to welding.

30 20. The method of claim 1, wherein the nanowires are created by a method selected from the group consisting of a vapor-liquid-solid process, a top-down fabrication method, a vapor-phase deposition process, a liquid-phase deposition process, and an electrochemical deposition process.

21. The method of claim 1, wherein the nanowires comprise any high-aspect ratio structure with a cross-sectional size of about 1 micron or

less and with variable cross sectional shapes, including a circle, ellipse, square, rectangle, hexagon and/or octagon.

22. A flexible and conductive thin nanowire film formed by the
5 method of claim 1, wherein the nanowires in the film have Ohmic junctions.

23. The flexible and conductive thin nanowire film of claim 22,
wherein an *I-V* curve of the film is substantially linear over a broad voltage
range.
10

24. The flexible and conductive thin NW film of claim 22, wherein a
volume density of Ohmic junctions meets or exceeds $1 \text{ } \mu\text{m}^{-3}$.

25. The flexible and conductive thin NW film of claim 22, wherein
15 the film is three dimensional and has a thickness of about 5 μm to about 10
 μm .

26. A nanowire film material, comprising:
a plurality of nanowires overlapping in a random orientation to form a
20 film material;

wherein the nanowires in the film material are welded at a point-of-
contact where two or more nanowires overlap to form an electrical
connection therebetween;

wherein the electrical connections between the plurality of overlapping
25 nanowires form Ohmic junctions.

27. The nanowire film of claim 26, wherein the nanowires are welded
at the point-of-contact by capillarity-driven surface diffusion.

28. The nanowire film of claim 26, wherein the film has a thickness
30 ranging from about the thickness of one nanowire to 100 μm or more.

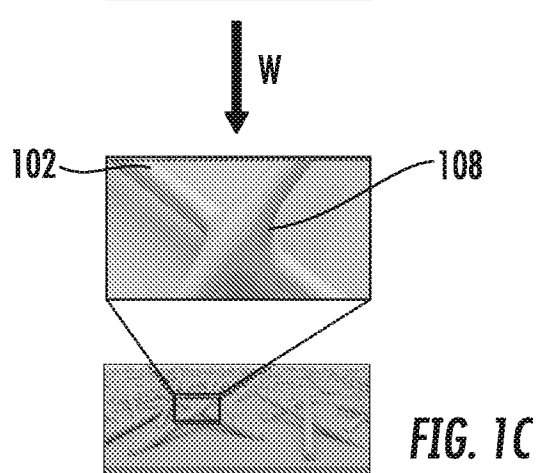
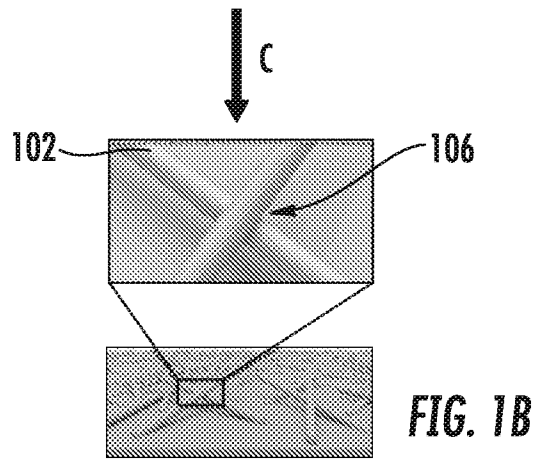
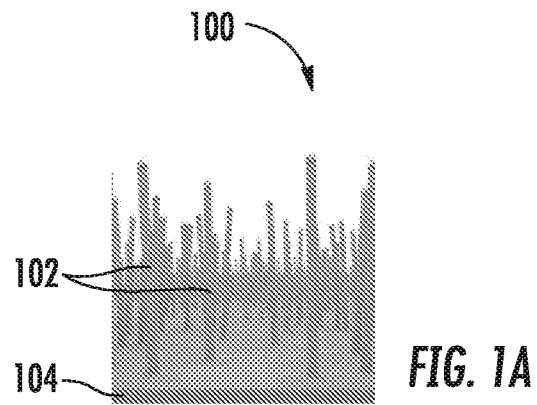
29. The nanowire film of claim 26, wherein an I - V curve of the film is substantially linear over a broad voltage range.

30. The nanowire film of claim 26, wherein a volume density of
5 Ohmic junctions meets or exceeds $1 \text{ } \mu\text{m}^{-3}$.

31. The nanowire film of claim 26, wherein the film is three dimensional and has a thickness of about $5 \text{ } \mu\text{m}$ to about $10 \text{ } \mu\text{m}$.

10

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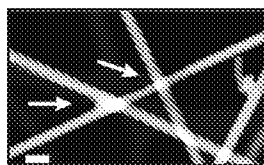


FIG. 2A

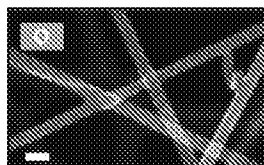


FIG. 2B

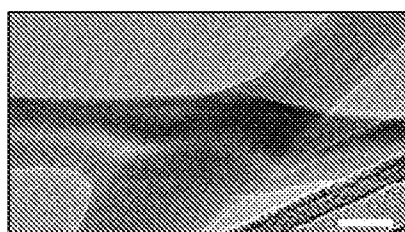


FIG. 2C

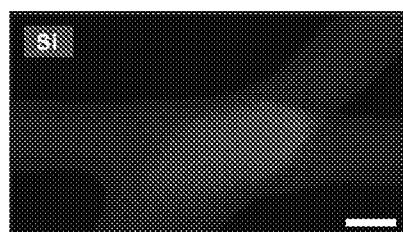


FIG. 2D

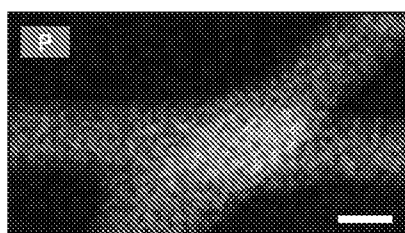


FIG. 2E



FIG. 2F



FIG. 2G

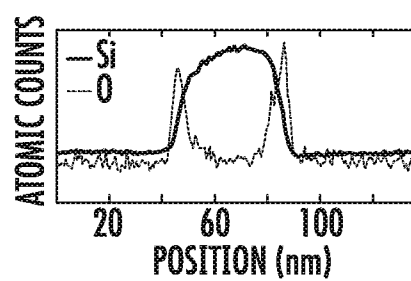


FIG. 2H

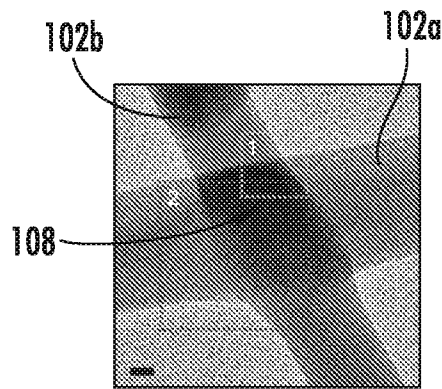


FIG. 3A

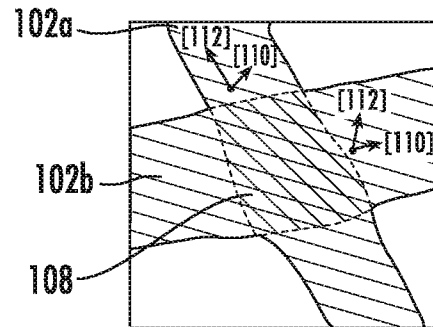


FIG. 3B

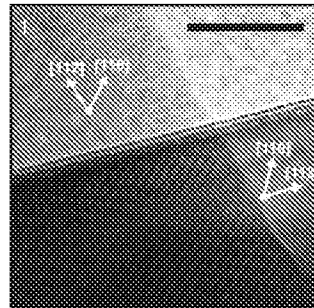


FIG. 3C

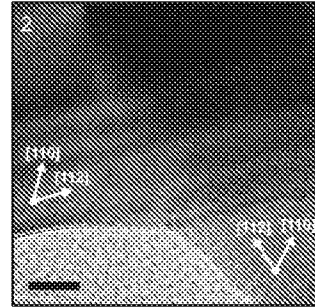


FIG. 3D

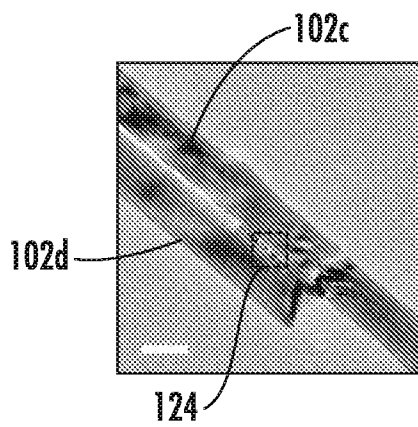


FIG. 4A

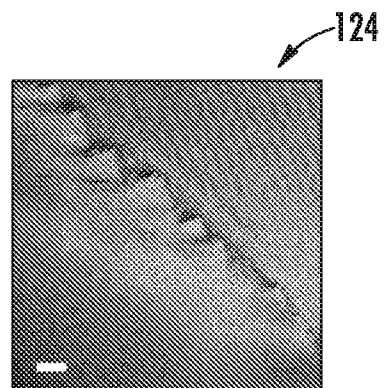


FIG. 4B

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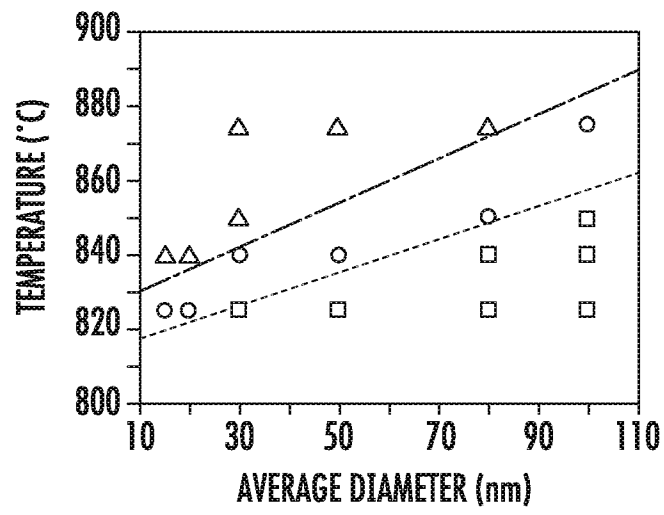


FIG. 5A

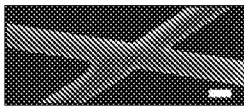


FIG. 5B



FIG. 5E



FIG. 5C

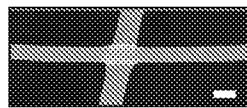


FIG. 5F

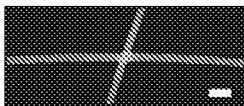


FIG. 5D

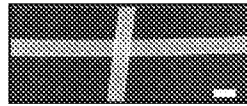


FIG. 5G

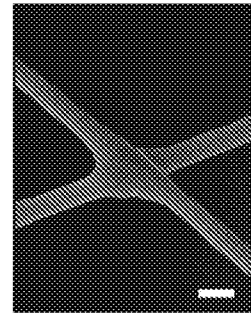


FIG. 5H

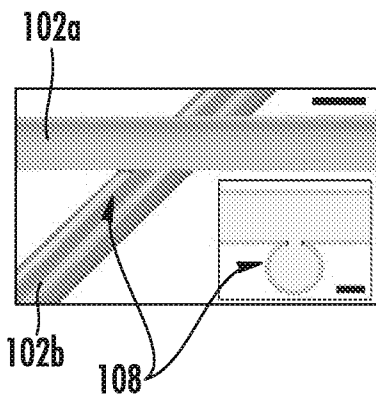


FIG. 5I

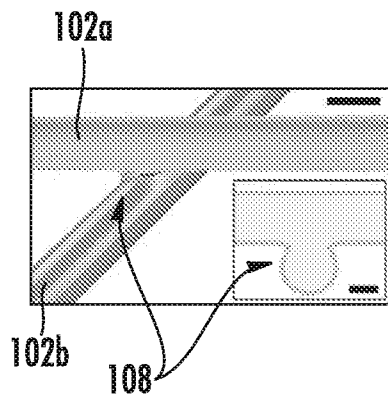


FIG. 5J

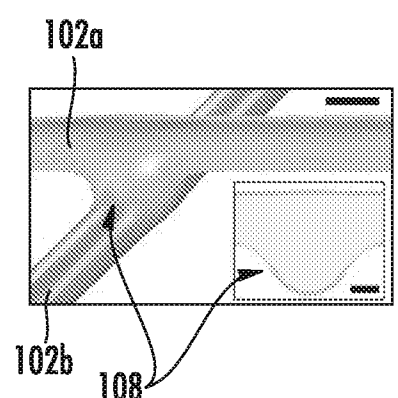


FIG. 5K

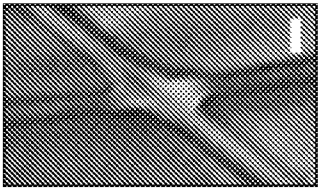
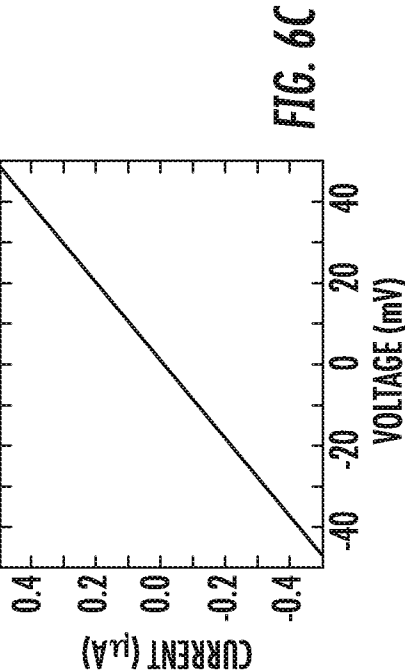
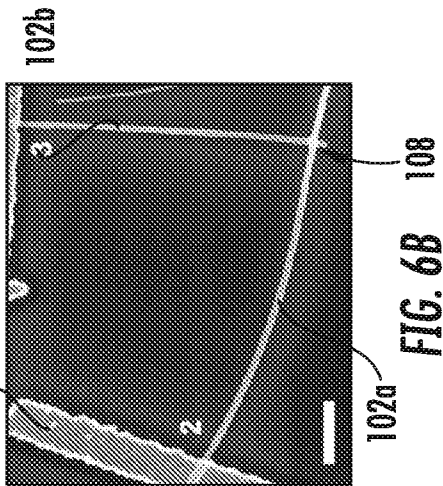
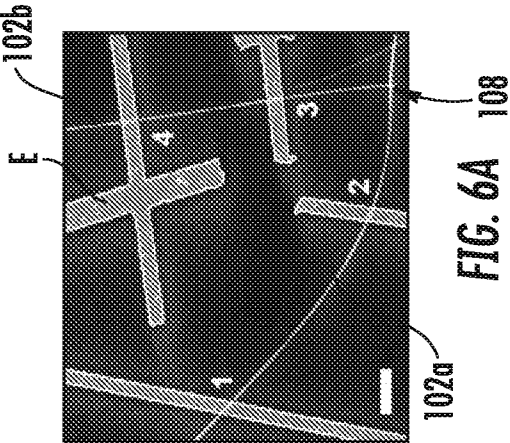
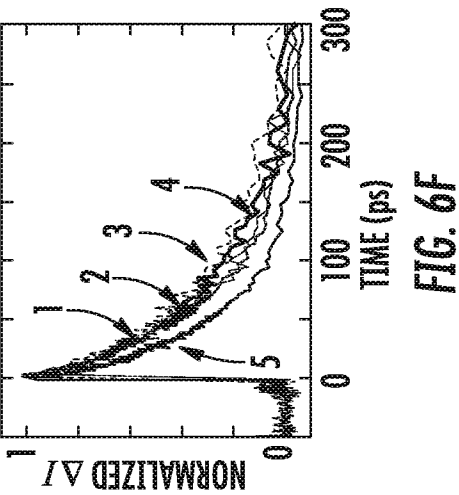
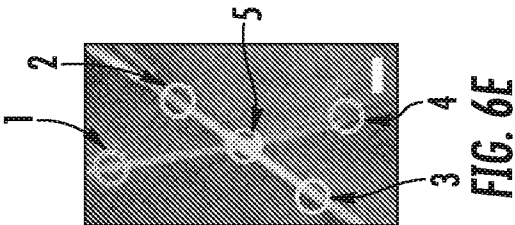


FIG. 6D



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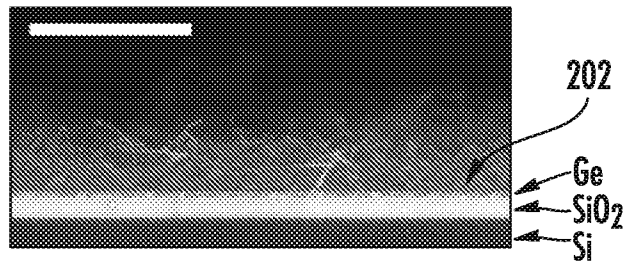


FIG. 7A

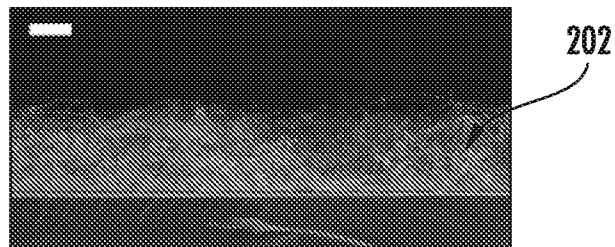


FIG. 7B

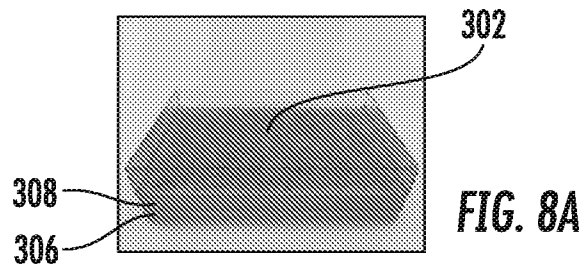


FIG. 8A

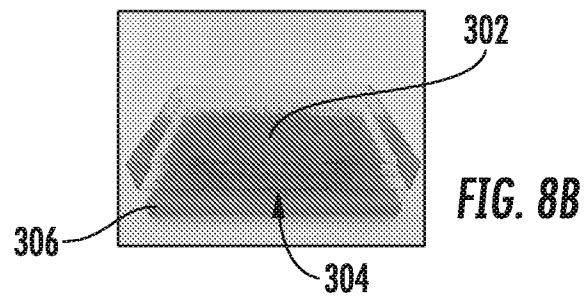


FIG. 8B

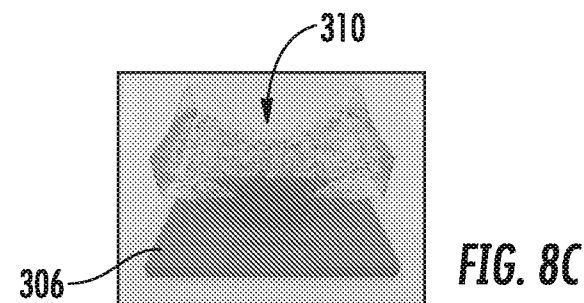


FIG. 8C

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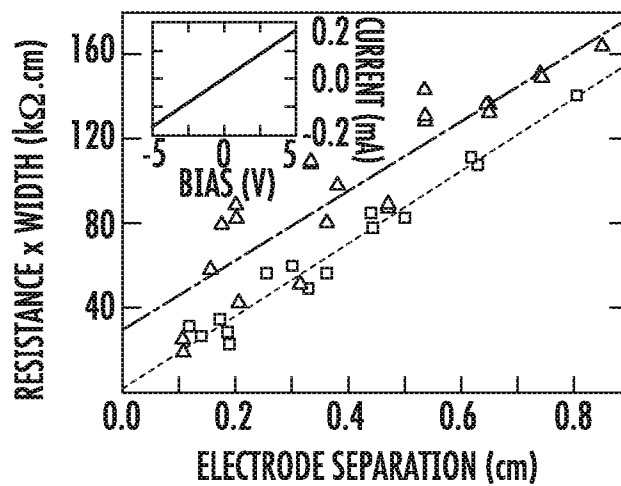


FIG. 9

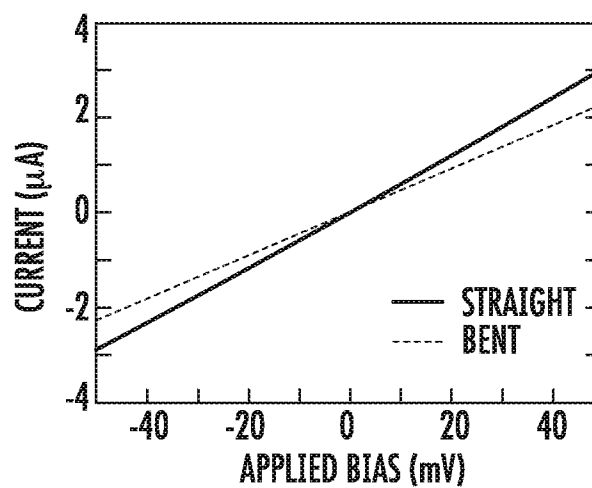


FIG. 10

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 2017/040362

A. CLASSIFICATION OF SUBJECT MATTER		
H01L 21/22 (2006.01) B82Y 40/00 (2006.01)		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols)		
H01L 21/00, 21/02, 21/04, 21/22, 31/00, 51/00, B82Y 40/00		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
PatSearch (RUPTO internal), USPTO, PAJ, Esp@cenet, DWPI, EAPATIS, PATENTSCOPE		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Zhiyong Gu, Hongke Ye, Diana Smirnova, David Small, and David H. Gracias,	26, 29
Y	Reflow and Electrical Characteristics of Nanoscale Solder, Wiley-VCH Verlag	1, 7-9, 11-12, 17-18, 20, 22-23, 27
A	GmbH & Co. KGaA, No. 2, 225 –229, 2006 [p. 227, 228, fig. 3, 5]	2-6, 10, 13-16, 19, 21, 24-25, 28, 30-34
Y	US 2006/0079455 A1 (RAMOT AT TEL AVIV UNIVERSITY LTD.) 13.04.2006, [0291], [0301], [0181], [0182], [0185], fig 10a	1, 7-9, 11-12, 17-18, 20, 22, 23
☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.		
* Special categories of cited documents:	"I" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family	
"A" document defining the general state of the art which is not considered to be of particular relevance		
"E" earlier document but published on or after the international filing date		
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)		
"O" document referring to an oral disclosure, use, exhibition or other means		
"P" document published prior to the international filing date but later than the priority date claimed		
Date of the actual completion of the international search	Date of mailing of the international search report	
23 October 2017 (23.10.2017)	02 November 2017 (02.11.2017)	
Name and mailing address of the ISA/RU: Federal Institute of Industrial Property, Berezhkovskaya nab., 30-1, Moscow, G-59, GSP-3, Russia, 125993 Facsimile No: (8-495) 531-63-18, (8-499) 243-33-37	Authorized officer M. Adireeva Telephone No. 8 499 240 25 91	

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 2017/040362

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	Zhiyong Gu, Hongke Ye, Adam Bernfeld, Kenneth J. T. Livi, and David H. Gracias, Three-Dimensional Electrically Interconnected Nanowire Networks Formed by Diffusion Bonding, Langmuir, 979-982, Vol. 23, No. 3, 2007 [p. 980 fig. 1, p. 981 col. 1, p. s2, lines 6-8, fig. 1, 3]	1, 7-9, 11-12, 17-18, 20, 22, 23
Y	US 2010/0193768 A1 (ILLUMINEX CORPORATION) 05. 08. 2010, [0038]	9, 20
Y	US 2016/0027846 A1 (PRESIDENT AND FELLOW OF HARVARD COLLEGE) 28.01.2016, [0160]	11