Biaxially stretchable transparent conductors that use nanowire networks

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Stretchable transparent conductors are required for flexible and wearable electronics. This study demonstrates biaxially stretchable transparent conductors that use silver nanowire networks. The use of buckled nanowire networks has previously been reported to lend stretchability to the transparent conductor in a single axis. However, a nanowire network that is prestrained and then buckled out-of-plane biaxially shows a deterioration of the electrical conductivity after a single cycle of stretching and releasing the strain uniaxially. This has been attributed to the loss of good electrical contact between the nanowires. By hot pressing the out-of-plane buckled nanowires to obtain an in-plane wavy nanowire network with good wire-to-wire junctions, a biaxially stretchable transparent conductor that maintains good electrical conductivity with stretching up to 10% is demonstrated. The methods of prestraining the nanowire network to achieve out-of-plane buckled nanowires and hot pressing the out-of-plane buckled nanowires to obtain an in-plane wavy nanowire network with fused junctions are expected to be practical for other classes of percolative networks based on one-dimensional (1D) materials used in flexible and stretchable applications.

I. INTRODUCTION

The transparent conductor is a crucial component in various optoelectronic applications such as light-emitting diodes, displays, touch screens, and photovoltaic devices. The conventional material used is a metal oxide such as indium tin oxide (ITO), which exhibits superb electrical and optical properties. However, ITO is brittle which limits its use in flexible and stretchable electronics. Stretchable electronics allows the integration of electronic devices on various types of stretchable substrates and liberates electronics from the conventional stiff silicon wafers. Hence, stretchable electronics realizes, for example, a better human-to-device interface. An increasing demand for stretchable electronics is expected as wearable electronics becomes more ubiquitous in our daily lives, as evident from the growing array of wearable health monitoring systems and wearable infotainment (e.g., Google Smart Watch, Samsung Gear Live, Moto360 smartwatch, Nike Fuelband, Jawbone UP). A display or touch screen (and hence transparent conductor) is often an essential component in such systems. Hence, various flexible/stretchable transparent conductors to substitute ITO need to be investigated.

Alternative materials include carbon nanotubes (CNTs),[1–11] graphene,[12,13] conducting polymers such as poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS),[14,15] nanowires[16–23] and various metal grids.[24–28] Stretchable transparent conductors can generally be formed by three different schemes: embedding the electrically conductive materials in elastic polymers,[1–9] depositing thin films of electrically conductive materials,[24–28] or creating wavy structures of electrically conductive materials on elastic substrates.[10–16,22,23]

Stretchable transparent conductors based on CNTs have been demonstrated by embedding CNTs in elastic polymers.[1–9] Stretchability is achieved because CNTs form a continuous conductive network in the elastic polymer even when the elastic composite is stretched. However, a high loading of CNTs, which is necessary to achieve high electrical conductivity, results in low optical transparency and high stiffness (i.e., poor stretchability) of the composites.

Thin metal meshes and films have also been explored as flexible and stretchable transparent conductors.[24,25,28] When a thin metal film on polymer substrate is stretched, cracks form in the metal film, and the electrical resistance increases steadily till the film ruptures.[28] This results in limited stretchability because the formation of cracks deteriorates the electrical performance and rupture occurs around 50% strain even when the metal film on polymer has been carefully designed.[28]

Another method to form stretchable transparent conductors, creating wavy structures of electrically conductive materials on elastic substrates, has been

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pursued using various electrically conductive materials such as CNTs,[10,11] graphene,[12,13] conductive polymers,[14,15] and metal nanowires.[16,22,23] As it is challenging to synthesize electronically uniform CNTs and large Schottky barriers exist at the junctions of dissimilar CNTs,[29] transparent conductors based on CNTs with high electrical conductivity and high transparency remain elusive. Graphene films also do not demonstrate high and stable electrical conductivity as there are often defects and grain boundaries in graphene films grown via chemical vapor deposition.[29] On the other hand, metal nanowire films possess high electrical conductivity (9–70 Ω/sq) and high optical transparency (90–96%).[16] As Ag nanowires are also highly ductile,[30–32] highly stretchable transparent conductors based on Ag nanowire films have been demonstrated.[16,22] Uniaxial stretchability of more than 100% has been demonstrated.[16]

Stretchability has been observed in one axis when the nanowire network is buckled in just one axis.[16] Nonetheless, biaxial stretchability is more practical for real world applications where electronics can be stretched and bent to a large extent in any direction the user desires. In an earlier work, we have formed two-dimensional (2D) in-plane buckled nanowire network by prestraining the elastic substrate in 2 perpendicular axes. We demonstrated that a nanowire network which is buckled in 2D as opposed to 1D is biaxially stretchable. However, the demonstrated biaxial stretchability without any increase in electrical resistance was limited to 5% strain due to the small degree of prestrain applied (constrained by the design of the stretch fixture).[22] In this work, we design a new stretch fixture that can prestrain the elastic substrate to a larger magnitude in 2D. The large magnitude of prestrain in 2D is found to result in a large extent of out-of-plane buckled silver nanowire network. The out-of-plane buckling is found to render a degradation of the electrical conductivity when the sample is stretched uniaxially. To our knowledge, this is the first time that 2D out-of-plane buckling in nanowire network is observed to degrade the electrical conductivity when the sample is stretched uniaxially. This study investigates this performance-limiting phenomenon (loss of electrical contact induced by Poisson effect) and provides a simple solution (mechanical hot pressing) so that large magnitude of stretchability (up to 10%) can also be achieved biaxially.

II. RESULTS AND DISCUSSION

A. Biaxially stretchable silver nanowire network

The fabrication procedure for creating a biaxially stretchable Ag nanowire film is shown in Fig. 1(a).

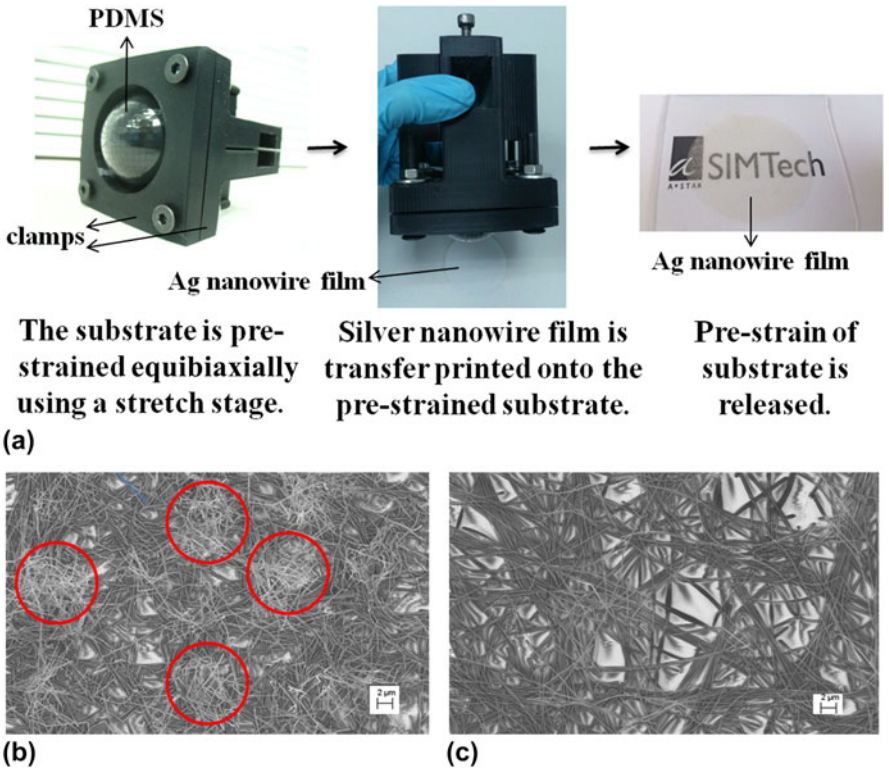


FIG. 1. (a) Schematic illustration of the fabrication of biaxially stretchable transparent conductor based on networks of silver nanowires (Ag NW) film. (b, c) SEM images of Ag nanowire films transfer printed onto a (b) prestrained and (c) non prestrained substrate, respectively. Selected areas (marked in red circles) of the Ag nanowire film in part (b) buckle out-of-plane.

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One weight percent of Ag nanowires (Nanotron Corporation) are dispersed in water. The length and diameter of the Ag nanowires are 49 ± 18 μm and 100 ± 20 nm, respectively. The polydimethylsiloxane (PDMS) substrates are prepared using the same method as described elsewhere.[22] A PDMS substrate is clamped on all sides and equibiaxially prestrained by pushing a sphere of 5 cm diameter against the bottom surface of the PDMS. The elastic PDMS is strained equibiaxially as the sphere pushes the thin PDMS out of its original plane. A silver nanowire film, which forms on a Whatman Anodisc membrane filter via vacuum filtration, is transfer printed from the membrane filter to the equibiaxially stretched PDMS. Upon complete transfer, the mechanical prestrain in the PDMS substrate is released by moving the sphere back into its original position. As the PDMS relaxes, the Ag nanowire film buckles out of plane of the PDMS substrate. This is evident from the SEM image shown in Fig. 1(b). Small distinct areas of Ag nanowires appear brighter (circled in red). Ag nanowires in these areas buckle out of plane (additional emission of secondary electrons along the buckled portions of the nanowires is responsible for the brighter appearance during imaging). Buckling is absent when the Ag nanowire film is transfer printed to a PDMS substrate that was not prestrained, as shown in the SEM image in Fig. 1(c). Waviness of the buckled nanowire films results from the compressive stress which the nanowires accommodate by bending when the prestrain is released. The electrical and optical properties of the Ag nanowire film were measured. The sheet resistance was ~50 Ω/sq and the transmittance at a wave length of 550 nm was ~70%. Lower sheet resistance with higher transparency can be achieved if longer nanowires were used.[16]

The use of buckled nanowire networks has previously been reported to lend stretchability to the transparent conductor in a single axis[16] or two perpendicular axes.[22] This is the first time that 2D out-of-plane buckling of nanowire network is observed when isotropic prestrain of 25% is applied on the elastic substrate. The biaxially buckled network that results is expected to lend stretchability in all axes of the two-dimensional plane. Interestingly, this is found not to be the case as shown in Fig. 2(a). The sample examined in Fig. 2(a) has been prestrained biaxially. The diameter of the circular film of Ag nanowires missing from the membrane filter after transfer printing from the membrane filter to the prestrained PDMS was measured to be 25 mm. This is compared to the diameter of the circular film of Ag nanowires transfer printed onto the PDMS substrate upon release of the prestrain: 20 mm. Hence, a prestrain of 25% has been applied in each axis of the plane. Figure 2(a) shows the normalized resistance (R/R_0) of the sample with applied uniaxial strain. R_0 and R are the electrical resistances before and after applying the uniaxial strain, respectively. The electrical resistance before applying the uniaxial strain is 53 ± 16 Ω. The normalized resistance at an applied uniaxial strain of 1, 3, and 5% increases by 1.1, 1.2, and 1.3 times, respectively. The normalized resistance further increases by 2.0 times (±0.3) when a 10% uniaxial strain is applied. Even upon releasing the uniaxial strain, the electrical resistance does not recover to its original value, unlike a reversible stretching process that was demonstrated in Ref. 33. The normalized resistance remains high at 1.8 times (±0.3) of the original value after complete relaxation. In a perfectly reversible stretching process, the nanowires return to be in good electrical contact again (and hence return to the original electrical resistance) after the strain is released.

The change in network percolation is observed under a scanning electron microscope (SEM) as uniaxial strain is applied to understand the stretching mechanism in a 2D out-of-plane buckled silver nanowire network, as shown in Fig. 2(b). The images on the left and right of Fig. 2(b) show the silver nanowire network when unstretched and stretched by 10%, respectively. Before stretching, the nanowires were buckled out-of-plane. As strain is applied, the wavy nanowires start to "flatten out" along the axis of the uniaxial strain and buckle more out-of-plane in the axis perpendicular to the uniaxial strain, as shown in the right image of Fig. 2(b).

B. Mechanism for loss of contact between nanowires during stretching

The question that arises then is why there is an increase in electrical resistance for 2D out-of-plane buckled films but not for films that are buckled in-plane, even though the nanowires "straighten out" during subsequent stretching in all cases. A film that has been biaxially prestrained by 25% (in this study) buckles out-of-plane. However, out-of-plane buckling was not observed in the film that was biaxially prestrained by 10% in an earlier work.[22] Only in-plane wavy nanowires were present.[22] Out-of-plane buckling in this study occurs due to the accommodation of larger compressive stress built up when the 25% prestrain is released. When a uniaxial strain is applied to the out-of-plane buckled nanowires, the nanowires that run approximately parallel to the stretch axis are stretched, as indicated by the red arrows in Fig. 2(c). In the transverse axis, the nanowires are compressed due to the Poisson effect of the PDMS substrate, as indicated by the blue arrows. This results in the stretched nanowires "flattening out" and the compressed nanowires buckling out of plane even further. Hence, the nanowires pull vertically, i.e., in the out-of-plane direction, away from each other and the number of nanowire-to-nanowire contacts decreases

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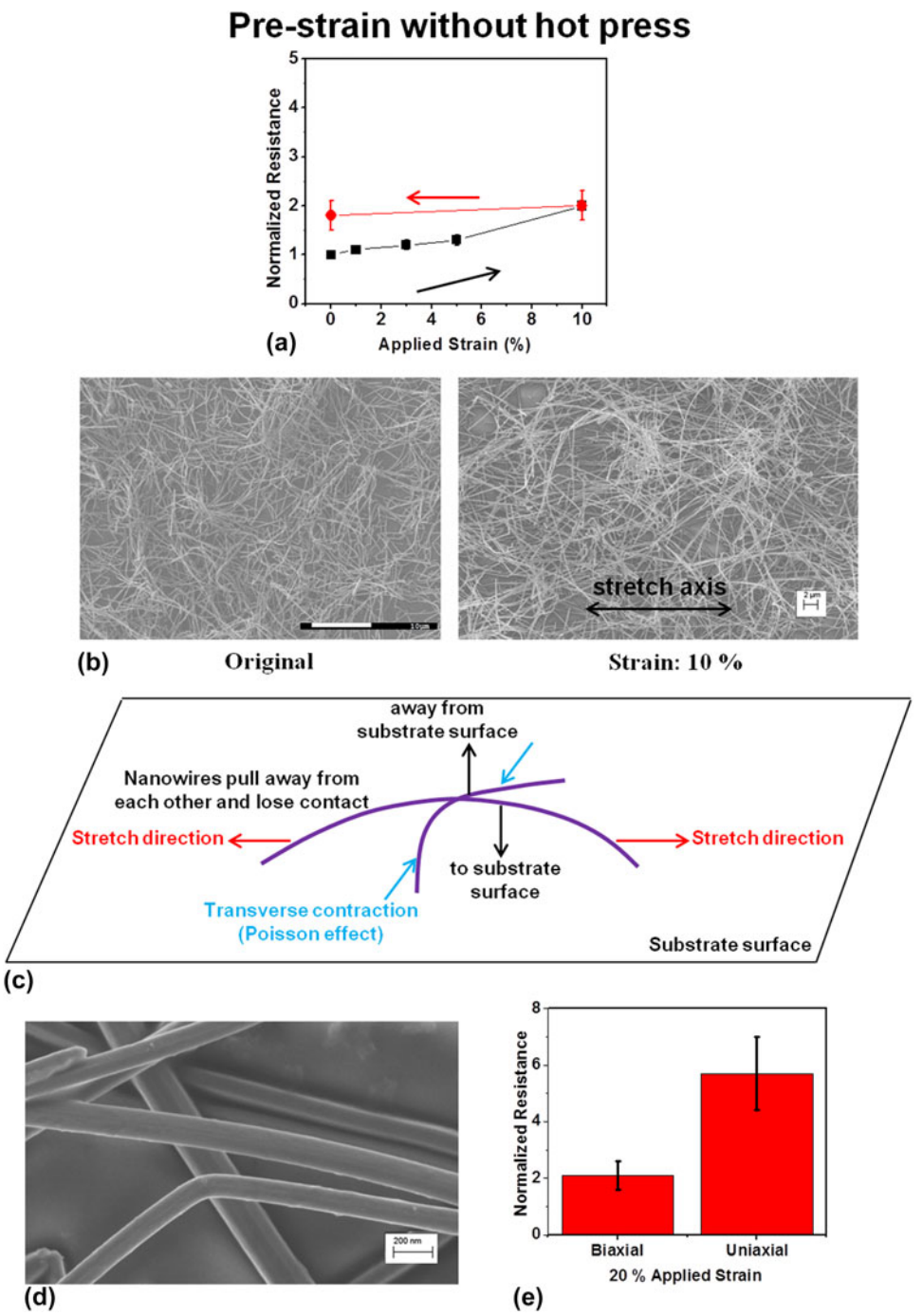


FIG. 2. Samples that have been equibiaxially prestrained but not undergone mechanical hot press: (a) Normalized resistance of the Ag nanowire films with applied uniaxial strain. The initial sheet resistance is 53 ± 16 Ω/sq. (b) SEM images of the Ag nanowire films upon stretching by 0 and 10%. The black arrow shows the direction in which the film was stretched. Scale bars in the left and right images are 10 and 2 μm, respectively. (c) Schematic illustration of stretching the equibiaxially prestrained Ag nanowire network in one axis. (d) SEM image of the Ag nanowire films. The nanowires are not fused at the junctions. (e) Normalized resistance of the Ag nanowire films with 20% applied strain. Samples that are strained uniaxially and biaxially are compared.

rapidly, resulting in an increase in overall electrical resistance. Figure 2(d) shows a SEM image of the nanowires. The nanowires are not fused to one another at their intersections and can easily move out of contact from one another.

To verify this phenomenon, the normalized resistance of the out-of-plane buckled nanowire network is compared when the sample is stretched 20% biaxially or uniaxially. Normalized resistance of the sample under 20% biaxial strain is lower than the normalized

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resistance when it is under 20% uniaxial strain [Fig. 2(e)]. The normalized resistance at 20% biaxial strain and 20% uniaxial strain is 2.1 ± 0.5 and 5.7 ± 1.3, respectively. This is in agreement with the phenomenon proposed above because the contraction in the transverse axis is absent in biaxial stretching. Nonetheless, a small increase in electrical resistance is also observed when the sample is strained 20% biaxially. This can be attributed to the loss of good contact at some of the nanowire junctions.

C. Maintaining good contact between nanowires during stretching

Pressing the out-of-plane buckled nanowires onto the substrate surface and fusion of nanowires at the junctions are expected to circumvent this problem of nanowires pulling away from one another. Various methods such as annealing[34–36] or mechanical pressing[21,37] have been explored to fuse nanowires at the junctions. By combining these two methods (applying a pressure of 4 MPa for 10 s at a temperature of 100 °C), we are able to (1) press the nanowires onto the plane of the substrate surface and (2) effectively fuse the nanowires together. Annealing alone is usually carried out at >200 °C to fuse the nanowires.[35,36] High annealing temperature is usually used because the presence of insulating residual poly(vinylpyrrolidone) (PVP: a byproduct of Ag nanowire synthesis) on the surface of Ag nanowires impedes the fusion of the nanowires.[34] As the polymeric substrate used in our devices is heat sensitive, we used a combination of mechanical pressing and gentle annealing at 100 °C (mechanical hot press). Mechanical pressing also presses the out-of-plane buckled nanowires onto the substrate surface. The mechanical pressure applied is relatively low at 4 MPa compared to 25 MPa[37] and 81 GPa[21] used in other works. Much higher mechanical pressure can potentially deform the PDMS substrate. Performance of the prestrained sample that has undergone mechanical hot press is vastly improved. Figure 3(a) shows the normalized resistance of the sample with applied uniaxial strain. The initial sheet resistance is 16 ± 4 Ω/sq. As uniaxial strain is increased up to 10%, normalized resistance remains pretty similar to before stretching. In fact, the normalized resistance barely changes even after 100 cycles of stretching to 10% [Fig. 3(b)].

Figure 3(c) shows the SEM images of the prestrained nanowire network that has undergone mechanical hot press with applied uniaxial strain. Unlike the samples that have been prestrained but not undergone mechanical hot

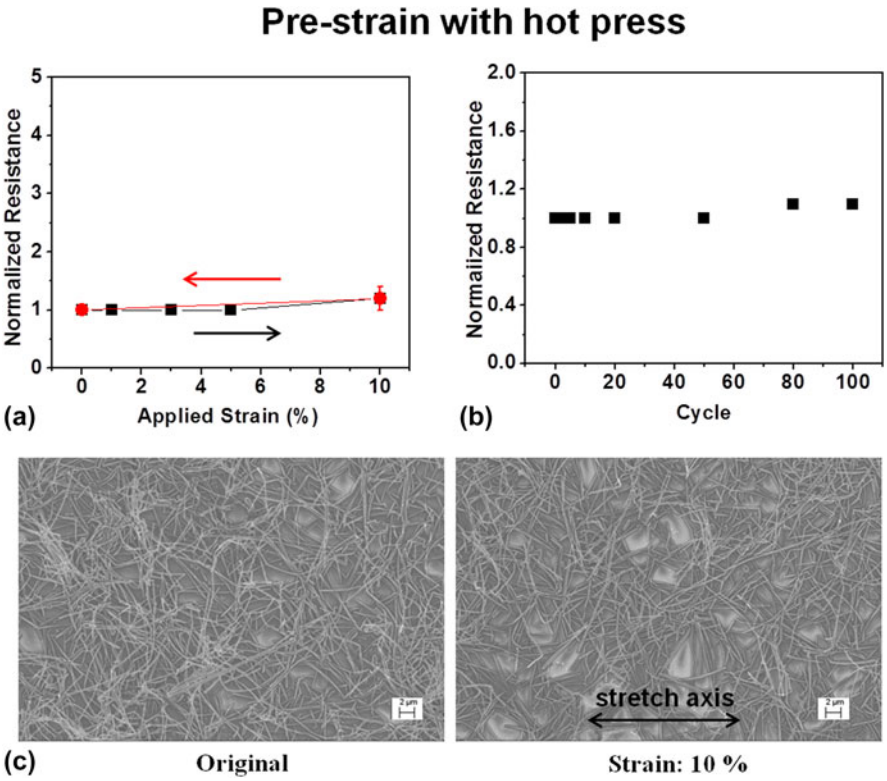


FIG. 3. Samples that have been equibiaxially prestrained and undergone mechanical hot press: (a) Normalized resistance of the Ag nanowire films with applied uniaxial strain. The initial sheet resistance is 16 ± 4 Ω/sq. (b) Normalized resistance of the Ag nanowire film as a function of the number of stretch and relax cycles. The applied strain was 10%. (c) SEM images of the Ag nanowire films upon stretching by 0 and 10%. The black arrow indicates the direction in which the film was stretched. Scale bars in the images are 2 μm.

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press, this network has been pressed onto the same plane. A flat transparent conductor film is also more desirable because it is essential in thin film devices such as thin film photovoltaic cells or light-emitting displays to prevent shorting of the devices. Although the nanowires in the network have been pressed onto the same plane, they remain wavy in-plane. As the strain increases from 0 to 10%, the nanowires in the network become less wavy in-plane but maintain electrical contact with one another [Fig. 3(a)].

As the nanowires are flexible, they are able to deform freely. This configuration is in principle similar to the serpentine microstructures used for stretchable electronics by Rogers and co-workers.[38–40] The group has carefully designed lithographically defined electrical interconnects with serpentine layouts which can bend and twist and improve stretchability. A random network of wavy nanowires can be perceived as a form of such interconnects but does not require expensive lithography because the nanowire dimensions are defined by their chemical synthesis process. However, the orientation of the nanowires with respect to one another is not well defined. Screen printing or spray deposition is expected to be able to define the dimensions of the nanowire network film as the nanowire network film is deposited from solution form. These methods enable large area printing at relatively low cost.

If only mechanical hot pressing without prestrain is performed on the nanowire network ("hot-press-only" sample), the normalized resistance also increases with applied uniaxial strain [Fig. 4(a)], despite the fusion between the nanowires in the network. A SEM image showing the fused nanowires in the hot-press-only sample is provided in Fig. 4(b). The normalized resistance at 3, 5, and 10% uniaxial strain is 1.3 ± 0.1, 1.7 ± 0.3, and 3.5 ± 0.8, respectively. The initial sheet resistance is 18 ± 8 Ω/sq. The increase in normalized resistance of the hot-press-only sample is higher than that of a prestrained-only sample. When the applied strain on the hot-press-only sample is released, the normalized resistance returns to 1.0, reflecting the reversibility of this process. In Fig. 4(c), one can observe the nanowires "stretching" in the direction of the strain axis as the hot-press-only sample is stretched. The source of the increase in electrical resistance of the hot-press-only sample with applied strain is examined next.

During stretching, the applied tensile stress cannot be accommodated by the straight nanowires in the hot-press-only sample, so the nanowires are strained and their junctions are forced to degrade. During stretching of a prestrained sample, the tensile stress is relieved when the nanowires flatten back onto the plane of the substrate. In the case of a prestrained sample that has subsequently undergone mechanical hot pressing, the nanowires are

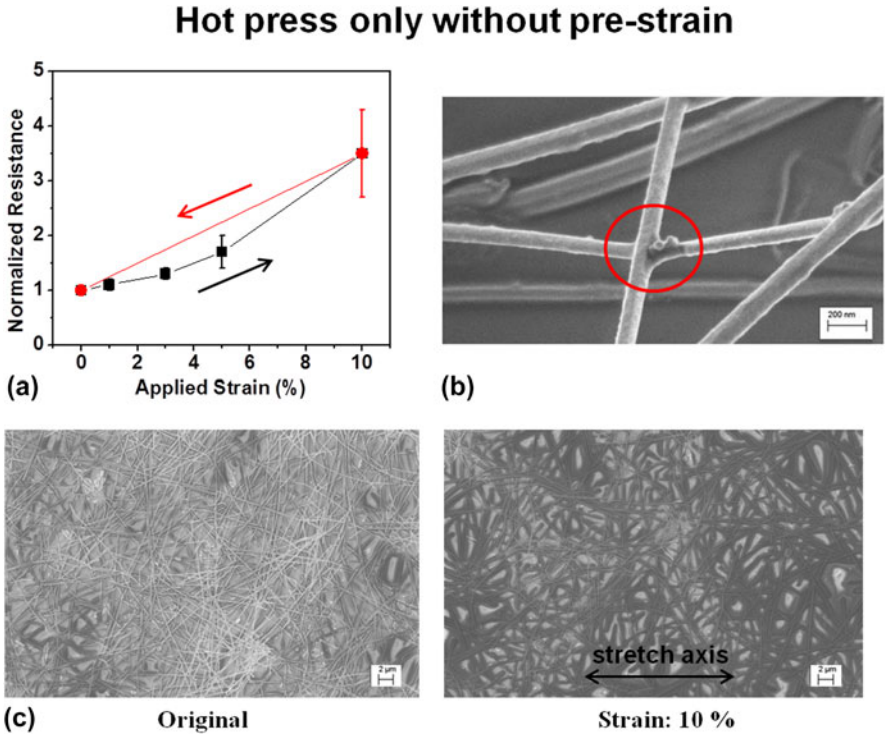


FIG. 4. Samples that have undergone mechanical hot press only: (a) Normalized resistance of the Ag nanowire films with applied uniaxial strain. The initial sheet resistance is 18 ± 8 Ω/sq. (b) SEM image of the Ag nanowire film after mechanical hot press. Fused junction is circled in red. (c) SEM images of the Ag nanowire films upon stretching by 0 and 10%. The black horizontal arrow indicates the direction in which the film was stretched. Scale bars in the images are 2 μm.

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very wavy (and they all lie on the same plane as the substrate). During stretching of such a sample, the wavy nanowires "straighten out". Hence, both prestrain (for creating buckled nanowires) and mechanical hot press (for pressing the out-of-plane buckled nanowires onto the substrate surface to form an in-plane wavy nanowire network and improved contact at the nanowire junctions) are essential for durable biaxially stretchable transparent conductors.

III. CONCLUSION

We demonstrate a biaxially stretchable transparent conductor that uses silver nanowire films. The principles established here are applicable to other biaxially stretchable conductors using 1D nanomaterials. The use of buckled nanowire networks has previously been reported to lend stretchability to the transparent conductor in a single axis. However, the same cannot be applied to a biaxially stretchable system that has out-of-plane buckled nanowires. This is because nanowires that buckle out-of-plane in a highly stretchable system and which are oriented in perpendicular axes pull vertically (i.e., in the out-of-plane direction) away from one another when strain is applied uniaxially. This results in a loss of contact between the nanowires and an increase in electrical resistance. A solution to this is found: By hot pressing the samples (100 °C at 4 MPa for 10 s), the out-of-plane buckled nanowires are pressed into an in-plane wavy nanowire network and the junctions between neighboring nanowires in the nanowire network fuse together. This method of mechanical hot pressing after prestraining is expected to be practical for other classes of percolative networks used for biaxially stretchable applications.

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