Flexible and Printed Electronics

PAPER

A highly deformable conducting traces for printed antennas and interconnects: silver/fluoropolymer composite amalgamated by triethanolamine

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Abstract

Stretchable conducting traces are the key component to realize wearable healthcare electronics; a conductor material that can withstand high strain conditions can be crucial. Here, we describe a simple fabrication pathway to achieve stretchable conductive ink for printing. Specifically, silver flakes and fluorine rubber are amalgamated by the aid of triethanolamine (TEA), which enhanced the compatibility of the components in solution state where methylisobutylketone is the co-solvent. Moreover, TEA plasticizes the composites after the printing and drying of the solvent, causing the composite to deform freely without losing conductivity. The composite exhibits a conductivity value of $8.49 \times 10^4$ S m$^{-1}$ at rest. The printed composite itself is not mechanically resilient after plastic deformation, but it has remarkable adhesion on elastomeric substrates. Thus, the printed ink on elastomers allows stretchable trace that can accommodate repeated stretching/releasing cycles. We fabricate and characterize stretchable printed antenna with three different designs (loop, patch, and bowtie) for the application of skin-adhesive electronics.

1. Introduction

The development of flexible and stretchable electronics relies heavily on the performance of conducting traces that can withstand high deformation while maintaining electrical conductivity [1, 2]. The stretchable trace is a key component to fabricate sensors and antennas for wearable electronics [3]. An important aspect for wearable healthcare electronics is the ability to accommodate mechanical strain and deformation correlated with body motion without deteriorating the performance of the electronics. Interconnects (i.e. conducting traces) are passive component in electronics and are easier to accommodate deformation compared to active components such as sensors or integrated circuits [4]. In fact, many system-level integration of stretchable electronics has been achieved by assembling macroscopic integrated circuit chips (mm to cm scale) that are encapsulated in stretchable substrate [5–7]. In this case, stretchable interconnects and antennas are the only two electrical components that are actually stretchable.

The printed stretchable interconnect can be achieved in numerous ways. Firstly, stretchability can be achieved even when the conducting material is not intrinsically stretchable. For example, metal films can be deposited on pre-stretched elastomeric substrate to achieve accordion-like ‘wavy’ structures [8]. Making structural relief in the metal patterns also allows stretchability, as can be exemplified by serpentine-[5], fractal-[9], mesh-[10] and coil-shaped [11] interconnects. These examples are not printed electronics, but their concepts can be easily applied in printing to achieve stretchability. Secondly, intrinsically stretchable, resilient, and conductive material can be developed. Percolating networks of nanowires embedded in or deposited on elastomeric substrate can be one example [12]. Metal precursor may be printed with
elastomeric host materials and then reduced as a post-treatment to achieve conductivity [13, 14]. However, the post-treatment often involves high curing temperature or corrosive chemical that may lead to the degradation of the host or the substrate materials. Thirdly, intrinsically plastic but non-resilient conducting material can be filled and then encapsulated in elastomeric trough, like a microfluidic channel. A liquid-metal filled polydimethylsiloxane (PDMS) channel can be a good example [15].

Enhancing printability to the conductive materials has been a challenging subject of research. For example, Someya et al introduced an imidazolium-based ionic liquid to achieve adequate viscosity of ink solution consisting of carbon nanotubes (CNT) and fluorinated rubber [16, 17]. This ink, however, has shown delamination issue between PDMS substrate and the interconnect layer. The group recently introduced a new formula that incorporates silver flakes, a fluorine rubber, and a surfactant to achieve enhanced adhesion on PDMS substrate [18]. Baik et al developed a printable silver and CNT’s composite ink with polyvinylidene fluoride binder, but a rather high curing temperature of 160 °C was necessary [19]. Yang et al developed a particle free conductive ink containing soluble Ag salt and adhesive rubber that allowed direct pen-writing [20]. This ink could maintain good adhesion with polyethylene terephthalate and polyimide, but multiple writing steps and chemical reduction post-treatment were necessary [20]. Pei et al developed a water based silver nanowire ink with cellulose and fluorosurfactant molecules [21]. Table 1 summarizes the key features of a few notable conductive inks for stretchable interconnects. An ideal ink of printable conducting material for stretchable interconnect will be to achieve all desirable traits, such as ideal viscosity for printability, adhesion to elastomeric substrates, high deformability of conductive material, and low curing temperature.

Here, we developed a new formula to achieve a stretchable ink with high conductivity. We introduced triethanolamine (TEA), which has conventionally been used as a surfactant in cosmetic industries or a plasticizer in plastic manufacturing [22, 23], in our ink formulation of silver flakes and fluorine rubber. When fabricating the ink, TEA facilitated dissolution of the components in the co-solvent of methylisobutylketone (MIBK), resulted in a homogeneous solution. The ink could be easily printed and adhered well on elastomeric substrates. After printing and drying of solvent, the composite exhibits high conductivity value of $8.49 \times 10^4$ S m$^{-1}$ without any need for post-treatment. Moreover, TEA plasticized the composite so that the printed traces were freely deformable without losing the conductivity. The composite was not mechanically resilient by itself, but a combination of stretchable substrate and the composite’s good adhesion allowed stretchable trace to be bended, twisted and stretched up to 100% without deteriorating its electrical and mechanical performances. We fabricated wireless local area network (WLAN) antennas with three different geometries: loop, patch, and bow-tie. The performances of three different antenna geometries were simulated and measured for original geometry and uniaxially-strained conditions.

2. Materials and methods

2.1. Materials

Fluorine rubber (DAI-EL 801) was purchased from Daikin industries ltd, Japan. 4-methyl-2-pentanone (or methylisobutylketone; MIBK) and Silver (Ag) flakes (10 μm, ≥99.9% metal traces) and TEA (≥99.0%) were purchased from Sigma Aldrich. A double sided clear transparent acrylic elastomer roll (VHB-4905) was purchased from 3M. All these reagents were used as-received without further purification.

2.2. Preparation and printing of elastic ink solution

The mixing ratio of the four components is a vital parameter which is responsible for both mechanical durability and electrical conductivity of the elastic ink. We identified the optimal weight ratio between the fluorine rubber:MIBK:TEA:the Ag flakes to be 1:2.3:1: X in order to achieve both stretchability and conductivity. The X values are 2.95, 2.40, 1.90 and 1.45 for 40 wt%, 35 wt%, 30 wt%, and 25 wt% Ag content, respectively. Here, the nomenclature of ‘wt% Ag content’ refers to the total weight of the solution including the solvent. Firstly, fluorine rubber dissolved in MIBK for 24 h. Then, TEA is added as a dispersive agent and the mixture was stirred for 6–8 h. Once the mixture becomes homogenous, silver flakes are added and the mixture was stirred it for 4 h to attain the silver elastic ink. All these procedures were carried out at the room temperature. The antennas (on-body loop, patch and bowtie) were patterned onto an elastomer substrate (VHB-4905) by using a 3D jet printer (nScrypt tabletop 3Dn printer). After printing, the antennas pattern were dried at 100 °C for 20 min to remove the excess solvent. When complete drying is needed, samples were placed in a vacuum oven for 24 h at 120 °C (Symphony-VWR, Vacuubrand 2 C). The schematic of the fabrication procedure is shown in figure 1.

3. Results and discussion

3.1. Mechanical durability of the ink

Using a combination of a tensile tester (Univert, Cellscale biomaterials testing) and a source meter unit (Keithley 2400), we studied the resistance change of printed traces during multiple stretch-release cycles. In one cycle, we tested the change in resistance until the trace’s ultimate failure at the strain value of ~500% (figure 2(b)). Here, we observed that the
### Table 1. Comparison study of various elastic inks.

| Components                                | Printing method  | Conductivity value at diff. strain %       | Curing temperature      | Reference |
|-------------------------------------------|------------------|-------------------------------------------|-------------------------|-----------|
| Sliver-CNT composite, PVDF                | Drop casting     | 5710 S cm$^{-1}$—0% strain                | 160 °C, 12 h            | [19]      |
|                                           | Hot rolling      | 20 S cm$^{-1}$—140% strain                |                         |           |
| Ag/Pt embedded                            | Screen printing  | 3012 S cm$^{-1}$—0% strain                | Room temperature, 24 h (drying) | [14]      |
| rGO mixed with PVDF                       | Hot rolling      | 322.8 S cm$^{-1}$—35% strain              | 150 °C, 90 min          |           |
| Ag flakes (91 wt%), polyurethane          | Screen printable| 3570 S cm$^{-1}$—0% strain                | 70 °C, 3 h              | [24]      |
|                                           |                  | 1200 S cm$^{-1}$—70% strain               | Low adhesion to PET/PVC |           |
| Ag flakes, MWNT, Benzyl mercaptan, Ethanol| Wet spinning     | ~6000 S cm$^{-1}$—0% strain               | 135 °C, 45 min          | [25]      |
|                                           |                  | ~260 S cm$^{-1}$—70% strain               |                         |           |
| Ag flakes, Fluorine surfactant            | Screen and stencil| 738 S cm$^{-1}$—0% strain                | 80 °C, 30 min           | [18]      |
| MIBK, Fluorine rubber                     | Printable        | 400 S cm$^{-1}$—70% strain                |                         |           |
| (Hydroxypropyl)methyl cellulose, fluorosurfactant, AgNW, Defoamer MO| Screen printed | 46 700 S cm$^{-1}$—0% strain | 150 °C, 30 min          | [21]      |
|                                           |                  | 8002 S cm$^{-1}$—70% strain               |                         |           |
| Silver flakes, TEA, 4-methyl-2-pentanone, Fluorine rubber (current study) | Printable | 849 S cm$^{-1}$—0% strain | Room temperature | Our work |
|                                           |                  | ~100 S cm$^{-1}$—110% strain              |                         |           |
resistance increases with respect to stretching is nearly linear up to $\sim 150\%$ of strain, whereas the rate of increase rapid grows at further degree of stretching. The operational repeatability of the printed film (figure 2(a)), it was stretched up to 50% strain at a crosshead speed of 0.3 mm sec$^{-1}$, and then the strain was released at the same rate (each cycle takes 50 s). This stretch-release cycle was repeated for 1000 times and the changes in the resistance were observed (figure 2(c)). The printed trace was conductive thought the test, but there was a progressing increase of resistance with respect to the stretch-release cycles. We hypothesize that the degradation is associated with local plastic deformation of the traces (such as small amount of thickness undulation at some local points); the exact mechanism is a subject of a future study.

3.2. Physicochemical properties of the conductive ink
Thermal degradation of the printed trace (fluorine rubber + TEA + Ag flakes; 0.72 is an important measure to assess maximum processing temperature after printing. We studied the property of the printed trace and three control samples using thermal gravimetric analyzer (TGA; TA Instrument Q500) in N$_2$ gas at a heating rate of 10 °C min$^{-1}$ from 30 °C to 700 °C (figure 3(a)). In all the samples, the solvent (MIBK) was thoroughly removed by vacuum oven drying at
120 °C for 24 h. The three control samples, TEA, fluorine rubber, and fluorine rubber + TEA (at the mixing ratio of 1:1), were selected to study the effect of each component and the influence of mixing. Fluorine rubber seemed stable up to ∼400 °C. TEA appeared to be volatile at temperatures as low as ∼170 °C. The reported boiling point of TEA is 335.4 °C and is non-volatile at room temperature (3.59 × 10⁻⁶ mm Hg), but the volatility rapidly increases at higher temperatures (for example, 10 mm Hg at 205 °C) [26]. Mixing with fluorine rubber significantly suppressed TEA’s volatility; even stronger effect was observed when mixed with Ag flakes.

Here, only 25% of weight loss was observed for the printed trace after the TGA cycle up to 700 °C. If more than 90% of TEA and fluorine rubber are to be volatilized at 700 °C, as shown in the result of the fluorine rubber + TEA, the projected weight loss is ∼60%. Interestingly, we observed the formation of black char residue in the printed trace sample after the TGA cycle, which suggest that there was some silver-mediated chemical reaction of fluorine polymer and/or of TEA. Further chemical analysis is required to elucidate this phenomenon.

Figure 3(b) shows the variation of conductivity of the printed trace with respect to the content of Ag flakes. The electrical conductivity increased monotonically with increasing Ag flake loading between 25 and 40 wt% of the weight of the total ink solution including the solvent. Here, the maximum conductivity value of 8.49 × 10⁴ S m⁻¹ was achieved in the 40 wt% Ag loading and minimum value of 4.10 × 10⁴ S m⁻¹ in the 25 wt% Ag.

3.3. Role of TEA in the elastic ink solution and in the stretchable traces
The elastic ink solution is composed of silver (Ag) flakes, fluorine rubber, an organic solvent (MIBK) and a dispersing agent (TEA). As shown in figure 4, the role of TEA in our stretchable ink is two-fold: (1) compatibilizer between the components to ensure a uniform dispersion of Ag flakes (filler) in the matrix of fluorine rubber in ink solution state and (2) plasticizer for the fluorine polymer network, which bestows high stretchability in printed conductive trace state.

TEA has long been used as an emulsifier to achieve complete wetting of water-soluble and oil-soluble ingredients in skin care and hair care products [27]. Due to the presence of 3 hydroxyl (−OH) groups and aliphatic chains on one molecule, TEA has also been used as a dispersing agent to disperse hydrophilic particles, such as titania [28] and hydroxyapatite [29, 30] in organic solvents for electrophoretic deposition and as a surfactant for the surface stabilization of nanoparticles [31, 32]. In our case, TEA functions as a dispersing agent by establishing compatibility between Ag flakes and MIBK which drives a uniform distribution in solution state. After drying of solvent, TEA contributes the formation of percolating conductive network of Ag flakes inside the polymer matrix of fluorine rubber. TEA also plays a role as a plasticizing agent. During the initial attempts to synthesize the stretchable ink solution, we found that the process of dissolution of fluorine rubber in MIBK was extremely time consuming. By the addition of TEA, the mixture dissolved homogeneously within few hours. This suggests that TEA diffuses into the fluoropolymer and quickly swell the network when in MIBK. In molecular sense, the compatibility may have been facilitated by as the matching between the partial polarity between the hydroxyl groups in TEA and the polar C–F bonds in fluorine rubber. The swelling of the fluoropolymer chains and their increased mobility can certainly facilitate the uniform dispersion of Ag flakes, whose surface is also compatibilized by TEA. The asymmetric molecular structure of TEA also aids in its high plasticizing ability [33], which gives high stretchability to the printed traces after drying out the solvent. Furthermore, very low vapor pressure of TEA (3.59 × 10⁻⁶ mm Hg at 25 °C) and high boiling point
ensures that TEA molecules do not evaporate in low-temperature operating conditions, which is an excellent trait for a plasticizer [23]. This point is supported by our TGA result in figure 3(a).

3.4. Application in stretchable antennas

Three types of stretchable antenna were designed and fabricated in this work. On-body stretchable antenna is designed to apply on the skin of human body while the other two are targeted to WLAN for internet of things (IoT).

3.4.1. On-body stretchable antenna

There are a few design considerations for antennas in on-body operation [34]. This is due to large relative permittivity of body tissues that causes significant electromagnetic wave loss. Person-to-person variation of shape and body composition complicates the problem to further extent [35, 36]. We attempted to have a first-order approximation model that resembles human arm to design on-body antennas by judicial simulation. Table 2 represents the parameters we employed to simulate human arm using known characteristics of body tissues and layers. The geometric model of the arm is drawn in figure 5(a). As can be seen the cross section is assumed to be ellipse while the dimensions of the main axes are presented in table 2 where A is the semi minor axis and B is the semimajor axis. Therefore, $A \times B$ is $4/\pi$ times the area of each ellipse. The length of the arm is also assumed to be 150 mm. The model includes skin, fat, muscle and bone. These values are used in Ansys HFSS full wave 3D electromagnetic simulator software to investigate the performance of the designed antenna. The mentioned parameters in table 2 are assigned to each layer in the software.

The loop antenna is one of the best options for on-body applications because the design’s magnetic dipole performance is relatively less affected by the relative permittivity of surrounding medium compared to other antenna designs [37]. Human body has very high relative permittivity because of its high-water content. For the on-body antenna, a square-loop structure with four circles is designed to operate at 3.5 GHz. The role of the four circles is to improve the gain and impedance bandwidth by enhancing the current distribution in the conductor path. The designed antenna structure was a 16 mm by 16 mm square and each circle is 4 mm in diameter while the thickness of the lines is 1 mm. For the substrate, a 1 mm thick acrylic elastomer VHB Tape 4905 (3M) with relative permittivity of 3.2 and tangent loss of 0.03 was used.

3.4.2. WLAN stretchable antennas

Using stretchable material and ink, reconfigurable antennas for many applications such as WLAN channels can be printed [38]. Finding new materials for stretchable antennas has been driven the progress of the field [39–41] and the pursuit for optimal material for better performance is still in progress. Figure 5(d) shows the stretchable patch antenna designed for WLAN applications. The patch design is one of the fundamental antennas. Here, a ground plane was included as a separate layer that is parallel to the antenna plane, whereas a dielectric layer laid between the two layers. The operation frequency of a patch antenna was directly depending on the patch length, thus stretching the patch altered its resonance.

Table 2. Dielectric and conductive characteristics of human body components used in our simulation.

| Layer | $\varepsilon_r$ | $\sigma$ (S m$^{-1}$) | Tan $\delta$ | $A \times B$ (mm$^2$) |
|-------|----------------|-------------------|-------------|------------------|
| Skin  | 38             | 1.4               | 0.28        | 45 $\times$ 64   |
| Fat   | 5.2            | 0.1               | 0.14        | 42 $\times$ 60   |
| Muscle| 52.7           | 1.7               | 0.24        | 38 $\times$ 54   |
| Bone  | 18.5           | 0.8               | 0.31        | 24 $\times$ 30   |

(335.4 C)
For the patch antenna, two-section impedance transformer was designed to match the reflection coefficient to $50 \, \Omega$. When the patch antenna was stretched in longitudinal direction ($Y$-axis), its operating frequency changed. The bowtie-slot antenna is a wide band monolayer antenna. When the bowtie antenna was stretched, its impedance increased.

The simulation and measurement results of $S_{11}$ in logarithmic scale for the three proposed antennas are shown in figures 5(b), (e) and (h), respectively for different stretching lengths. The $S_{11}$ graph versus frequency shows how much power is reflected back from the antenna input port. When more power is accepted by the antenna, less power is reflected back thus the radiated power is higher. In dB scale, the lower value means lower reflected power. The operational bandwidth of the antenna is defined for frequency range where the reflection coefficient is lower than $-10 \, \text{dB}$, i.e., more than 90% of the power is accepted by the antenna. The frequency correlated to the lowest depth in the $S_{11}$ graph is considered as the resonance frequency. By stretching these antennas, the operating frequency was lowered.

The on-body antenna is designed to have the resonant frequency at 3.5 GHz at the original length. The solid blue curve in figure 5(b) is the simulation result of the on-body antenna at its original length. The depth of $S_{11}$ curve happens at 3.5 GHz and the level is
around –12 dB which means the antenna accepts more than 93% of the fed power at this frequency. The measurement of S11 depicted as the solid black curve with circle in figure 5(b) shows around 98% accepted power at the same frequency. While the 100% stretched length, performance is studied using the simulation and measurement shown as red dash-line and solid green with triangle in figure 5(b), respectively. Both show around 99% accepted power at 1.75 GHz which is half the original resonant frequency. It was expected that by increasing the length of the antenna twice of its original length the resonant frequency will be halved [42].

The patch and bowtie-slot antennas have similar performances in terms of S11 as can be seen in figures 5(e) and (h), respectively. The patch antenna was designed to have resonance at 5.5 GHz. The solid blue and dash-green curves in figure 5(e) illustrate the simulation and measurement results of the input reflection coefficient (S11) of the patch antenna for the original length. Both curves show more than 96% of power acceptance by the patch antenna at 5.5 GHz. Then the patch antenna was stretched in y-direction for 32% and 65%. The operating frequency of a patch antenna depends on the patch length in y-direction so by increasing the length of patch it is expected that the resonant frequency lowers.

The simulation and measurement of the input reflection coefficient (S11) for 32% stretched length are depicted with solid-red having circles on and dash-pink having circle on in figure 5(e), respectively. By increasing the length of the patch 32% more than its original length it is expected that the resonant frequency decreases 23% which is 4.1 GHz. The same result has been performed in both the simulation and measurement. Similarly, if the patch is stretched 65% more than the original length the operating frequency decreases 40% which 3.3 GHz. The solid black curve with cross marker and dash-brown with triangle marker in figure 5(e) represent the simulation and measurement result of the input reflection coefficient. The simulation shows more than 98% accepted power while in the measurement only 95%. The bowtie-slot antenna is designed to operate at 5.3 GHz at its original length.

The solid blue and dash-green curves in figure 5(h) demonstrate the simulation and measurement of the input reflection coefficient (S11) of the bowtie-slot antenna at its original length. Both curves show more than 99% accepted power by the antenna at the desired frequency. The resonant frequency of a bowtie-slot antenna also depends on its total length in y-direction. Similar to the on-body and patch antennas, the resonant frequency of a bowtie-slot antenna proportional to the inverse of the electrical length of antenna [43]. By increasing the length of the slot 42% more than its original length it is expected that the resonant frequency lowers to 4 GHz while stretching to 110% more than the original length should lowers it to 2.5 GHz. The simulation and measurement results for the input reflection coefficient (S11) of 42% and 110% stretching for the bowtie-slot antenna are shown with solid-red with circle marker, dash-pink with circle marker, solid black with cross marker, and dash-brown with triangle marker in figure 5(h), respectively. The input reflection coefficient results show proper agreement between the simulation and measurements for all three antennas. The discrepancies are due to the changes in the thickness of the substrate and conductor in practice and also slight decrease in the conductivity and increase of the dielectric constant of substrate which were not included in the simulations [44].

The simulation and measurement results of the radiation patterns in the E-plane and H-plane are illustrated in figures 5(c), (f) and (i) respectively. The E-plane of an antenna is the plane containing the electric field vector, which is the radiation pattern in the YZ plane in this paper. The H-plane of an antenna is the plane containing the magnetic field vector, which is the radiation pattern in the XZ plane. θ is the polar angle between Z axis and the line crossing the origin and φ is the azimuth angle in the XY plane between X axis and the line crossing the origin. These patterns are presented as normalized gain in dB scale for better comparisons. The radiation pattern is the graphical illustration of how power is radiated in the space. The higher value shows that the antenna radiates better at that specific angle, thus needs less power to communicate properly with other antennas at a certain distance.

The bowtie design is another fundamental antenna. The bowtie design complements the dipole configuration by radiation due to the electric current flowing around the edges of the slot pattern (see figure 5(g)). For the bowtie design, only one conductor layer is required (i.e. there is no ground plane as a separate layer, which is necessary in the patch antenna). Therefore, its fabrication by printing is straightforward. The shape of the slot is similar to a bowtie and it is fed from the center. The bowtie-slot antenna is a wide band monolayer antenna. The design of the stretchable bowtie antenna can be seen in figure 5(g). The simulation and measurement results for the input reflection coefficient for different stretched length versus frequency and radiation patterns are presented in figures 5(h) and (i), respectively.

4. Conclusion

Here we have shown a novel synthesis formula for highly deformable conducting traces, where TEA is used as a compatibilizer and a plasticizer for the composite of fluoropolymer and Ag flakes. The printed conducting traces on elastomeric substrates showed an excellent combination of electrical and mechanical properties. We discussed the role of TEA
in the ink solution and in the printed traces. Using the conducting traces, we fabricated a 3.2 GHz on-body loop antenna, as well as WLAN patch and bowtie antennas. These antennas were stretchable and their resonance frequency dropped as the antenna was stretched. These results show that our novel ink can be a promising candidate as a conductor material for wearable electronics and IoT applications.

**Author contributions**

AK, T-GL and H-JC conceived ideas on materials; AK, T-GL and H-JC fabricated and characterized materials and fabricated antennas. HS, MMH, HAD, T-GL, HC, and H-JC fabricated and characterized antennas. AK, T-GL and H-JC wrote the manuscript.

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