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3D Printed Functional and Biological Materials on Moving Freeform Surfaces

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Abstract

Conventional 3D printing technologies typically rely on open-loop, calibrate-then-print operation procedures. An alternative approach is adaptive 3D printing, which is a closed-loop method that combines real-time feedback control and direct ink writing of functional materials in order to fabricate devices on moving freeform surfaces. Here we demonstrate that the changes of states in the 3D printing workspace in terms of the geometries and motions of target surfaces can be perceived by an integrated robotic system aided by computer vision. A hybrid fabrication procedure combining 3D printing of electrical connects with automatic pick-and-placing of surface-mounted electronic components yielded functional electronic devices on a free-moving human hand. Using this same approach, cell-laden hydrogels were also printed on live mice, creating a model for future studies of wound-healing diseases. This adaptive 3D printing method may lead to new forms of smart manufacturing technologies for directly printed wearable devices on the body and for advanced medical treatments.

Keywords

3D printing; wireless electronics; feedback control; robotics; bioprinting

Experimental details for procedures and parameter settings can be found in the Supporting Information.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

All animal studies were approved by the Institutional Animal Care and Use Committee (IACUC) at University of Minnesota.

Competing financial interests

The authors declare no competing financial interests.

The creation of seamless interfaces between biological systems and multifunctional devices is a vital step towards the fabrication of next-generation wearable devices for applications in teleoperation,^[1,2] medical treatments,^[3,4] health monitoring,^[5–7] and even personal entertainment^[8–10]. Commercial wearable devices are either rigid (e.g., smart fitness watches), or soft but with determinant shapes (e.g., haptic gloves for virtual reality applications). Such properties are often incompatible with target biological surfaces that possess highly arbitrary, dynamically varying geometries, such as skin and tissues. As a result, non-seamless interfaces between wearable devices and target surfaces can be created, compromising user comfort and device performance, such as inaccurate capture of physical status and incomplete exchange of cells at the interface.

One approach toward fixing this problem is to fabricate a flat device with thin, deformable materials using microfabrication, and subsequently transfer them to the target surface for a conformal interface. This approach has been highly successful in creating flexible, stretchable, and biointegrated electrical circuits,^[2,11–14] sensing arrays,^[7,15,16] energy harvesting devices,^[17,18] digital displays^[9,19] and optogenetic controllers.^[20] Alternatively, we suggest that devices can be autonomously fabricated without the need for microfabrication facilities in freeform geometries that are actively adaptive to target surfaces in real time, driven by advances in multifunctional 3D printing technologies. With recent progress in printable functional materials and device designs, 3D printing is not only capable of replicating shapes in both macro and micro scales,^[21] but also interweaving a diverse palette of functional, biological, and biocompatible materials. This approach has been used for a variety of applications, such as regenerative pathways,^[22] microbial wearables,^[23] fully 3D printed quantum dot light-emitting-diodes (QD-LEDs),^[24] and electromagnetic structures.^[25] High-performance discrete electronics could also be integrated into the 3D printed structure to yield hybrid devices.^[26]

3D printing is thus an attractive manufacturing method due to its unique versatility to directly fabricate freeform functional devices that adapt to the geometries of target surfaces. Previous approaches relied on open-loop calibrate-then-print procedures by utilizing cumbersome reverse-engineering techniques, with demonstrations such as 3D printed tactile sensors on a model hand,^[27] microfluidic devices on whole organ models,^[28] and bacteria-derived materials on a doll face.^[29] Yet, these approaches were only applicable to static target surfaces. To enable adaptive 3D printing on moving freeform surfaces, a closed-loop feedback system is required to allow real-time correction of printing errors introduced by the dynamically changing workspace.^[30] Indeed, previous attempts to track and ink-jet print on a moving human hand have been carried out.^[31,32] Our approach advances these technologies further by: (1) compensating for the full six degrees of freedom of rigid body motions; (2) being applicable to more general tasks with arbitrary shapes of the target surface; (3) being compatible with a continuous, extrusion based printing method in order to achieve a faster speed of fabricating 3D structures and to make use of a diverse palette of functional materials; and (4) demonstrating the printing of functional electronic devices as a proof of concept.

Here we report a new method, termed adaptive 3D printing, for the fabrication of multifunctional devices on moving freeform surfaces. The motivation is to establish a

workflow for on-body devices that is autonomous, requires minimal human intervention, and is ambient-processable without the requirement of being tethered to a microfabrication facility. Specifically, this method integrates a closed-loop feedback control system onto a compact and portable extrusion-based 3D printer. The concept is illustrated in Figure 1. The geometric information of the target surface is first acquired in the form of a dense point cloud via a structured light scanner (Figure 1a). The sampled geometric information is then combined with a real-time estimation of the rigid-body motion of the target surface (Figure 1b), and as a whole fed back to the motion controller for adaptive 3D printing. During printing, a discrete electronic component, such as a surface-mounted LED, is first pick-and-placed onto the target surface automatically (Figure 1c). Functional inks such as conductive and insulator inks are directly printed onto the moving target surface to create electronic circuits (Figure 1d). The printed devices are finally dried at room temperature and powered by a wireless transmission system (Figure 1e).

A key component of the process is the development of functional inks which meet criteria related to conductivity and ambient processability. Specifically, important design criteria for conductive inks used in the adaptive 3D printing of electronic devices are: (1) the viscosity of the ink should be tunable while maintaining self-supporting structures; (2) the ink solvent should evaporate quickly such that the device becomes functional on the same timescale as the printing process; (3) the printed electrodes should become highly conductive under ambient conditions. In particular, if biological surfaces are involved, high temperature sintering should be avoided.

To fulfill these criteria, we developed a highly conductive, water-soluble ink by adding silver flakes (10 μm , Sigma-Aldrich)^[33] as conductive fillers to poly(ethylene oxide) (PEO, $M_v = 1,000,000$ Da), with water and ethanol as solvents. Using the high molecular weight PEO as a matrix afforded a relatively high viscosity and good mechanical strength in the final material. The viscosity of the ink was tailored by varying the content of PEO in the solutions from 5 to 15 wt% (Figure 2a). All inks exhibited a typical shear-thinning behavior, which is desirable for controllable extrusion during 3D printing.^[34] A high shear modulus at low shear stress helped the inks retain their filamentary form after printing (Figure 2b). The content of PEO was optimized to be 12 wt%, considering the high shape retention at various printing resolutions (Figure 2c) and a reasonable extrusion speed (~ 7 mm/s) to reduce print time.

To decrease the ink drying time, ethanol was added to increase evaporation speed. As ethanol content increased from 0 to 80 wt%, the resistance of the printed electrodes reduced by 90% within 10 min (Figure 2d). This is a crucial feature of the inks, since a short printing time on a living subject is desirable. However, the ethanol is limited by the solubility of 1,000,000 Da PEO (Figure S1). Thus, the solvent ratio of water:ethanol was 1:4 in the final ink formulations. The viscosity of the ink with higher ethanol content was slightly decreased (Figure S2), because the lower density of ethanol led to a higher ink volume.

The content of silver flakes determined the electrical conductivity of the ink, which exhibited a typical percolation threshold behavior (Figure 2e). A power-law theory used to fit the data was given by^[35]

$$\sigma = \sigma_0(V_f - V_c)^s \quad (1)$$

Here σ is the electrical conductivity of the dried inks, σ_0 is the power law constant, V_f is the silver volumetric fraction, V_c is the filler critical volume fraction at the percolation threshold, and s is the critical exponent. From our experimental data, V_c and s were found to be 0.257 and 0.771, respectively. The conductivity of the printed electrodes was enhanced by their anisotropic nature (Figure S3), with the cross-sectional and top view SEM images of the filament shown in Figures 2f and 2g, respectively. The shear forces induced by the high extrusion pressure (2–3 MPa) aided in the alignment of the silver flakes along the printing direction,^[26,36,37] resulting in conductive pathways in the silver-PEO network that improved the conductivity of the printed trace. The ink composed of 90 wt% Ag exhibited the highest conductivity of $(1.38 \pm 0.0814) \times 10^4 \text{ S cm}^{-1}$, which was only one order of magnitude smaller than that of bulk silver. The ink with 90 wt% Ag exhibited desirable mechanical performance as well due to the high molecular weight of PEO, showing more than 300% strain at break (Figure S4). The yield stress decreased from 7.6 to 4.3 MPa when the Ag content increased from 75 to 90 wt% (Figure 2h). In a cyclic tensile test, viscoelasticity was observed, with a time delay in returning to the original shape (Figure S4d). The portion of the specimen that was under plastic deformation extended in size during cycling, shifting the stress-strain curves. The ink maintained good adhesion to artificial skin (Figure S5a) after 1,000 cycles of bending at a radius of 52 mm (Figure S5b). Local buckling of the printed trace was observed when the cyclic bending radius reduced to 32 mm (Figure S5c). Similar phenomena were also observed upon repetitive stretching of human skin at the metacarpophalangeal joint, as an extreme deformation case compared to the skin on the back of a hand (Figures S5d–g). These mechanical properties facilitated the printed devices on human skin.

Overall, the optimal ink formulation was comprised of 12 wt% PEO in the polymer solution, 20 wt% water in the solvent composition, and 90 wt% Ag in the dried ink. Since the bending induced from skin or joint movement by the human body may affect the performance of the printed devices,^[38] the electrical behavior of the bended filaments was examined. As shown in Figure 2i, the change in resistance decreased to 10% when the bending radius of the specimen increased to 12 mm, a typical value of human body movement.^[39] These data suggested that the conductive ink with fast drying speed, high conductivity, proper mechanical strength, and stable resistance under deformation could yield flexible printed electrodes for wireless devices on skin.

To demonstrate the performance of the conductive ink in wireless electronic devices, inductive coils were fabricated to power automatically pick-and-placed LEDs (Figure 3a), with the image of a final device shown in Figure 3b. The wireless device was fabricated layer-by-layer (Figure S6a–d), consisting of an inductive coil at the bottom, an insulator layer in the middle, a crossover conductive trace at the top, and a surface-mounted LED as the electrical load of the circuit. Given constraints on the outer diameter and spacing of the inductive coil, electrical characteristics of the circuit were tunable by varying the number of

coil turns (Figure S6e–i). For instance, the resonance frequency of the device could be tuned to fall into the operation range of frequency according to the design of the wireless power transmission circuit (Figure 3c; Figure S7a). Although an increasing number of coil turns was more preferable in terms of higher inductance and thus higher generated electromotive force (Figure 3d), the side effect of the increment of impedance resulted in more power loss (Figure S7b). This trade-off was reflected in the Q factors of different coil designs (Figure S7c).

In addition to powering a wireless device, the 3D printed inductive coil can also be used as a wireless moisture sensor which shared the same coil design of the previously introduced wireless powered device. When immersed into water, the printed conductive traces underwent a reverse process of drying, thus exhibiting higher impedance. Under controlled moisturization, the ink is capable of maintaining the printed form (Figure S8a,b), and recovering to the initial impedance level after drying, with an error of $3.9 \pm 14.1\%$ ($n = 3$). The change of impedance in the inductive coil was reflected in the shift of frequency response from the readout system (Figure S8c) that was inductively coupled with the 3D printed wireless sensor. Different moisture states of the conductive traces were imparted by applying water vapor with a constant flow rate to the wireless sensor for different periods of time. The change of resonance frequency and impedance responses from the readout coil indicated different moisture states of the conductive traces (Figure 3e). The reusability of the wireless sensor was studied by repeatedly moisturizing the device and letting it dry ($n=4$). The moisturized conductive trace recovered to a conductivity level that was close to the original dry state (Figure S8d).

Next, to directly fabricate a wearable wireless device on a moving human hand, an adaptive 3D printing platform was developed. This consisted of a delta robot, monitor cameras for long-distance observation of printing states, and tracking cameras mounted on the robot end-effector for precise localization of the target surface (Figure S9). A delta robot was utilized due to its faster travel speed, larger carrying load, more compact size and affordable cost relative to typical gantry-like systems. We chose a computer-vision-based method for tracking rather than using off-the-shelf depth sensors, in order to achieve customizable tracking precision, resolution and processing speed to match the varying 3D printing scenarios. The spatial fidelity of the printed structure on a moving surface depends on three system specifications: (1) diameter of the extrusion nozzle; (2) image resolution and processing errors in the visual tracking system; (3) resolution and accuracy of the 3D printer. Since the dimension of the extrusion nozzle is submillimeter, the latter two specifications becomes dominant in determining the resolution limits in this current setup. To characterize the tracking performance, the printer nozzle was commanded to track a given trajectory on a linear stage with programmed reciprocating motion, which was “unknown” to the adaptive printing platform (Figure S10a). The tracking error could be controlled to be under 1.5 mm on target surfaces exhibiting slow motion (<8 mm/s) (Figure 3f), referring to the speed profile of the arbitrary hand motion captured by the visual tracking system (Figure S10b). By further increasing the moving speed of the printer, the tracking error could maintain the same accuracy for target surfaces with faster motion (10 mm/s), though sacrificing the tracking stability of static targets (Figure S10c). To achieve optimal printing accuracy in real

applications, the moving speed of the printer could be parametrized based on prior knowledge of the speed range of the target surface.

As a further demonstration of the adaptive 3D printing platform, we directly fabricated wirelessly powered electronic devices onto an unconstrained human hand (Figure S11). The surface-mounted LED was automatically pick-and-placed onto the target surface via an empty nozzle under controlled vacuum levels (Movie S1). Since there are not abundant visual features on a human hand suitable for computer-vision-based tracking, fiducial markers were attached before printing for robust feature detection (Figure 4a; Movie S2). To compute the geometrically adaptive toolpath for each layer of the device and also register the spatial information of the LED and the fiducial markers, the hand was 3D scanned into a dense point cloud with a structured light scanner (Figure 4b; Movie S3).

During printing, the 3D position and orientation of the hand were estimated via a Perspective-n-Point (PnP) method based on the positions of the fiducial markers detected by the tracking cameras (Figure S12), with an average refresh rate of 20 Hz. The pose information of the hand was then fed back to the printer controller in real time to compute the toolpath to be followed by the end-effector, which was adaptive to both the geometry and the motion of the human hand (Figure 4c, Movie S4). The hand was unconstrained during printing and could move and rotate randomly within the range of the 3D printing workspace ($\phi 230 \times 270$ mm) (Figure 4d; Movie S5), with a linear speed below 8 mm/s, so that the motion-induced printing errors were tolerable for device functionality. After drying under room temperature for 15 min, the functionality of the 3D printed wireless device was demonstrated by lighting the LED with a wireless power transmission coil (Figure 4e; Movie S6). Wireless moisture sensors were also fabricated on human hands via the adaptive printing platform. When the 3D printed wireless moisture sensor was placed under the readout coil with the skin on the back of the hand in dry and wet states, distinguishable frequency responses from the readout coil were observed (Figure 4f).

The ease of removal of wearable devices is an important feature, particularly for those interfaced onto biological surfaces. Our studies showed that the interfacial bonding between the skin and ink was sufficient to support the printed form of the devices on the hand for more than two hours without detachment. Indeed, it was observed that the pulling force required to overcome the adhesion of the device resulted in a significant amount of deformation of the detached trace (Figure S13). However, the devices could be simply removed without destructive deformation by peeling them off the skin with a tweezer (Movie S7). The high molecular weight of PEO in the conductive ink helped maintain a continuous trace during the peel-off process (Movie S8). If devices need to be removed for the purpose of recycling the surface onto which they are printed, such as the surfaces of rubber gloves or clothes, it was found that the printed devices could be degraded by first dissolving the PEO in water and then physically removing the remaining detached silver particles (Movie S9).

Finally, for future biological applications of the adaptive 3D printing process, its utility was demonstrated in the study of printing cells on moving targets as a proof-of-concept for treating wound-healing diseases. Recessive dystrophic epidermolysis bullosa (RDEB) is a

genetic disease which results in life-altering blisters and open wounds. Advances have been made through genetic correction of the mutation causing the disease, but an appropriate *in vivo* delivery system for the gene-edited cells has yet to be found. Adaptive 3D printing could present a natural solution. Before adaptively printing cell-laden hydrogels into the wound bed of live mice, preparation procedures including attaching fiducial markers (Figure 4g) and 3D scanning (Figure 4h) were performed. The mouse was sedated before printing but still underwent motions such as breathing and twitching, which was compensated by the adaptive printing system. The presence of viable cells was detected via bioluminescence imaging following 4 hours of printing (Figure 4i), showing the potential of the adaptive printing method to retain the functionality of the cell-laden ink. This procedure presents a unique opportunity in the future to study the wound-healing capabilities of gene-corrected RDEB cells via delivering them in an autonomous manner that could allow for re-epithelization in a biologically relevant environment.

To summarize, we report a new adaptive 3D printing method for the autonomous manufacturing of multifunctional devices on moving freeform surfaces, which combines direct ink writing with closed-loop feedback and computer-vision-based control techniques. Wirelessly powered devices and wireless moisture sensors were fabricated on human hands, via 3D printing a novel functional, fast-drying ink that is highly conductive, room-temperature processable and flexible. The adaptive 3D printing method also enabled autonomous deposition of cell-laden hydrogels onto desired locations of live mice. Future studies will focus on the following: 1) Optimizing the adaptive printing system to reduce printing time and improve printing fidelity for fine and more sophisticated electronics, 2) integrating multifunctional electronic components in the wirelessly powered devices for active bio-machine interfacing; 3) optimizing the moisture sensor as a sweat sensor for wireless monitoring of physical exertion or stress levels; 4) printing layered structures of gene-corrected cells as skin substitutes, for *in vivo* studies on improved wound healing; 5) applying adaptive printing to insects and animals for large-scale production of cyborgs as self-distributed data collectors in ecosystems. Overall, by incorporating a diverse palette of high quality electrical and biological materials with the adaptive 3D printing platform, new possibilities will be created in the wearable device industry, in biological and biomedical research, and in the study and treatment of health conditions.

Experimental Section

Materials System:

The polymer solution was prepared by dissolving PEO ($M_v \sim 1,000,000$, Sigma-Aldrich) in a dual solvent system comprised of water and ethanol. The conductive ink was prepared by adding silver flakes ($10 \mu\text{m}$, Sigma-Aldrich) into the PEO solution, followed by mixing in a planetary mixer (ARE-310, Thinky) for 15 min and centrifuging for 5 min to remove air bubbles.

Rheology Measurements:

The rheological properties of the inks were characterized using a rotational rheometer (ARES-G2, TA Instruments) equipped with 25 mm parallel plates and a solvent trap cover. The measurements were carried out with shear rates from 0.001–1000 s⁻¹.

Tensile Test:

The specimens were prepared by printing various inks in the form of rectangular plates (AGS100 Gantry System, AeroTech, Inc.). The tensile tests of the specimens were carried out on a dynamic mechanical analyzer (RSA-G2, TA Instruments) at 0.1 mm s⁻¹.

Cyclic Bending Test: The artificial skin (Deep-layer Suture Training Pad, Laplay) was bent as a simply supported beam, with the two ends fixed to a linear stage (ANT130-XY, Aerotech, Inc.) and a lab jack, respectively. The linear stage was controlled to undergo cyclic lateral motion with different amplitudes and a constant speed of 25 mm/s.

Conductivity Measurements:

The resistances of the filaments printed with the ink were measured with four-point resistivity probing equipment (S-302, Signatone) connected to a source meter (Keithley 2450, Tektronix). To measure the change of conductivity under different bending radii, filaments of the conductive ink were bent to be conformal to the 3D printed test fixtures, with the resistance measured by a source meter.

Drying Time Measurements:

Filaments connecting two copper electrodes were printed on a glass microscope slide (Corning). Right after printing was finished, the resistance of the filament was measured continuously with a source meter for 30 min.

Characterization of Inductive Coils:

The inductance and impedance of the printed coil were measured by an RF impedance analyzer (4291B, Hewlett Packard). The resonance frequency of the printed coils was measured with the wireless method,^[40] which was also used to characterize the sensing behavior of the printed coils. The printed coils were moisturized with a humidifier (Model EE-5301W, Crane). The recovery states of the devices were measured by moisturizing for 2 min and drying in ambient environment for 10 min.

Wireless Power Transmission System:

The wireless power transmission system was powered by a DC power supply (E3611A, Agilent). The DC signal (1.4V, 0.18A) was converted to an AC signal by an oscillator circuit consisting of a power transistor (D40C7, Central Semiconductor Corp.) and an output inductive coil. During wireless power transmission, the output inductive coil was placed on the top of the wireless device printed on the hand with a clearance of 10–20 mm.

3D Scanning:

The target surface was scanned by a structured light scanner (HDI 109, LMI Technologies). The raw scan data was post-processed by a geometry editing software (DAVID 3, Hewlett Packard) to yield a uniform, smooth mesh.

Adaptive 3D Printing:

Adaptive 3D printing was executed by a 3D printer (Delta Rostock 3D Printer, Anycubic). Visual tracking was enabled by two machine vision cameras (Chameleon3, FLIR) mounted on the end-effector of the 3D printer. The extrusion of material was controlled by a digital pneumatic regulator (EFD) that was connected to a syringe (EFD) mounted on the end-effector of the 3D printer. The overall adaptive printing software was custom-programmed in Python.

Bio-printing on mice:

Superficial wounds were created on 4–6 week-old Crl:NU(NCr)-Foxn1nu mice (Charles River, 490). A squamous cell carcinoma (SCC) GFP/Luc line was mixed with the bio-ink and printed onto the wounded mice with the adaptive printing platform. Mice were imaged on day 0 with a Xenogen IVIS 100 imaging system (Xenogen Corporation).

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Additional Information

An Institutional Review Board (IRB) assessment at the University of Minnesota determined that the experiment involving 3D printing on the hand of a subject does not meet the federal definition of human subjects research and, therefore, a formal IRB review was not required.

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Figure 1.

Adaptive 3D printing of multifunctional devices. a,b) Schematic images of a) 3D scanning of the target surface to be printed on, and b) real-time tracking of the rigid-body-motion of the target surface. c-e) Schematic images of c) pick-and-place of discrete electronic components on the target surface using a vacuum nozzle, d) direct writing of conductive ink, and e) demonstration of powering the LED via a wireless power transmission system.

Figure 2.

Development of the conductive ink with proper printability, high conductivity and flexibility.

a) Apparent viscosity as a function of shear rate for the inks with different PEO contents (wt %). b) Shear moduli as a function of shear stress for various inks. c) Printed ink traces with different resolutions. d) Plots of R/R_t over time immediately after inks were printed with various solvent compositions (wt%). R_t is the resistance of the ink dried in ambient environment for 30 min. e) Electrical conductivity of the dried inks as a function of silver flake content ($n = 6$). The solid line is a power-law fit to the data using Equation 1 (R-square = 0.806). f) Cross-sectional SEM image of the ink filament with 90 wt% Ag, showing the alignment of Ag flakes induced by shear force from the printing process. g) Top view SEM image of the ink filament with 90 wt% Ag. h) Yield stress of the inks as a function of Ag content ($n = 4$). i) Change of resistance of the ink filament with 90 wt% Ag as a function of bending radius (R-square = 0.977).

Figure 3.

Characterization of the wireless devices and the adaptive printing system. a) Layout of the layered design of the 3D printed wireless device that powers a surface-mounted LED. b) Photograph of the printed wireless device. c) The impedance responses and d) the inductances of coils with varying number of coil turns ($n = 3$). e) Impedance response of the printed inductive coil performing as a wireless moisture sensor when exposed to water vapor for different periods of time. f) Tracking error of the adaptive printing platform as a function of various moving speeds of the target surface ($n = 2000$), with a printer moving speed of 14 mm/s.

Figure 4.

Demonstrations of adaptive 3D printing of functional electronic and biological materials. a) Image of fiducial markers placed around the automatically pick-and-placed LED on a human hand. b) Images of the 3D scan result of the human hand and the geometrically adaptive toolpaths for each layer of the device. c) Plot of the trajectory of the randomly moving hand and the adaptive toolpath of layer 1, both computed in real-time by the visual tracking system. d) Image of adaptive 3D printing of the wireless device on a human hand that can move freely in the workspace. e) Image of powering the LED in the printed device via a wireless power transmission system. f) Impedance responses from the readout coil (inset) due to the change of moisture level of the surface of the human hand. g) Images of the live CrI:NU(NCr)-Foxn1nu mouse without (top) and with (bottom) an artificial wound and fiducial markers. h) Image of the 3D scan data of the live mouse and a zoom-in view (inset) of the scanned wound bed. i) Bioluminescence image (Day 0) of the mouse with printed squamous cell carcinoma (SCC) GFP/Luc line (Left) and the control group (Right).