Characterizing the Electrical Properties of Anisotropic, 3D-Printed Conductive Sheets for Sensor Applications

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Abstract—This paper introduces characterization techniques to investigate electrical properties of 3D-printed conductors. It presents the combination of a physical model to describe frequency dependent electrical properties of 3D-printed conductors; the use of infrared thermography in combination with Joule heating to characterize electrical anisotropy in 3D-printed sheets; and the use of the voltage contrast scanning electron microscopy method (VCSEM) to determine potential distributions in 3D-printed sheets. By means of lock-in thermography, infrared (IR) measurements are improved and amplitude modulation enables lock-in thermography at excitation frequencies above the thermal cut-off frequency. Measurements on sensor samples show the potential of the methods for characterizing sheet-like, conductive structures. The characterization methods allow improvement of 3D-printed sensor designs and exploit electrical properties of 3D-printed conductors.

Index Terms—3D-Printing, Conductive, Anisotropy, Infrared Thermography, Voltage Contrast Scanning Electron Microscopy, Sensor Characterization.

I. INTRODUCTION

3D-PRINTING conductors, and in particular sensors, by means of fused deposition modelling is an upcoming, promising area of research [1], where 3D-printed piezo resistive, EMG and capacitive sensors have been demonstrated [2], [3] and a significant amount of research has been done on electrical properties of conductive-polymer composites for printing [4], [5]. However, printing conditions affect the electrical properties due to voids and bonding conditions between adjacent traxels (i.e. track-elements produced by the printing process), as shown by measurements and simulations [5], [6] and affect the performance of 3D-printed sensors [3]. On the other hand the printing conditions can be used to 3D-printed strain sensors with adjustable sensitivity and anisotropy [7]. Insight is gained by developing appropriate physical models, representing conductive structures by fused traxels. In previous research conductance was already described in this way; however this was limited to a 1D-solution in the DC-case [8]. Furthermore, electrical characterization has primarily focused on global impedance measurements, whereas the 2D distribution of the electrical impedance in sheets is expected to give important additional insights. To do so two non-invasive techniques are studied that can measure in one go the distributed conductive properties and which potentially can be used to study both DC and AC characteristics. Here we show that IR thermography, so far used for studying heating power in 3D-printed samples [5], can also be used for studying the anisotropic electrical properties of 3D-printed samples as it has been done for the characterization of carbon fiber reinforced polymers [9], conductive textiles [10], micro-irregularities in electronic components [11], [12] and to image networked conductive nanomaterials [13]. In this paper we also show that the voltage contrast scanning electron microscopy method (VCSEM), used e.g. to characterize
conducting networks in carbon nanotube composites [14] and used for semiconductor failure analysis [15], can be applied to 3D-printed conductors too. For the first time IR thermography and VCSEM are applied to conductive 3D-prints in combination with a model to study the distributed electrical properties of 3D-printed conductors and sensors.

**II. METHODOLOGY**

**A. Modeling**

In short the methodology consists of 3D-printing and measuring of single layer, multi-traxel sheets. A voltage is applied over the sheet. Various characterization methods are used to experimentally observe the impedance of the sheets, the potential voltage distribution or the Joule heating induced temperature profiles. Two different 3D-print designs are used for the main experiments, with leads on the opposite or parallel sides (Fig. 1). The two geometries are used to study if the characterization methods are able to distinguish voltage and power dissipation distributions for a current path which is primarily on one side of the sample versus the situation for a current path crossing the sample.

In order to drive the experiments and to test our understanding of the structures the conductors are simulated by means of traxels with bulk properties (resistivity $\rho$, relative permittivity $\varepsilon_r$) and mutual contact properties (inter-traxel resistivity $\sigma$, inter-traxel capacitance $C_0$) to describe the electrical characteristics. This makes the model an AC-variant of the work by Hampel et al. [8]. An equivalent electrical network to represent these properties is shown in Fig. 2. The traxel resistance $R_T$ and traxel capacitance $C_T$ represent the lumped bulk properties (the combination of $\rho$ and $\varepsilon_r$ with the traxel geometry), whereas the inter-traxel resistance $R_{IT}$ and inter-traxel capacitance $C_{IT}$ represent the mutual contact properties (the combination of $\sigma$ and $C_0$ with the traxel geometry). For this study focus is mostly put on the DC behaviour, primarily considering the resistive components. The structures are simulated by the Finite Element Method (FEM) using the Electric Currents module of COMSOL. The voltages and power dissipation density are simulated as well as the impedance between the leads, for comparison with the experimental results. The electrical properties are implemented through the material properties and contact impedance functionality. The COMSOL simulations are validated by means of a mesh convergence study, showing convergence of the impedance for finer meshing. The error of the total resistance for the default mesh size with respect to the ultrafine mesh is below 0.1%. The model is used as a qualitative comparison with the experiments, to study isotropic versus anisotropic conduction. Depending on the magnitude of $\rho$ and $\sigma$ the current will mainly follow the bulk (isotropic conduction) or meander with the traxels (anisotropic conduction) and anything in between.

**B. Fabrication**

CAD designs (Solidworks, Dassault Systèmes) are processed in slicing software (Simplify3D). A flexible carbon black-filled TPU (Palmiga Innovations PI-ETPU 85-700+ [16]) is printed using a consumer grade multi-material 3D-printer (Creator Pro, Flashforge Corporation) with a dedicated extruder for flexible materials (Flexion Extruder, Diabase Engineering). A 0.6 mm nozzle is used with the default extrusion width multiplier of 1.2. The ETPU is printed at 210 °C with a bed temperature of 50 °C. The samples are single-layer sheets of 15 mm by 15 mm by 0.2 mm, with 19 traxels per sample with a traxel width of approximately 0.8 mm. They are printed on glass wafers with electrical contact leads on either the same (parallel) or opposite side (Fig. 1). The contacts are made via copper tape with Ag-conductive paint (Electrolube SCP26G) and enamelled copper wires (to prevent out-gassing problems of typical polymer wire insulation in SEM experiments).

**C. Resistance Methodology**

Resistance measurements are carried out using an LCR-meter (HP 4284A) in a four-terminal configuration. The measured total resistance is a combination of the traxel and inter-traxel resistances. The impedance of the model is used to fit the combination of $\rho$ and $\sigma$ on the total resistance. The measured VCSEM voltage distribution subsequently gives the information to visually fit the ratio of the values of $\rho$ and $\sigma$ in the simulations. In this way a similar shape for the voltage distribution can be obtained, which then yields unique values for $\rho$ and $\sigma$. In earlier research impedance measurements were performed with an LCR-meter in four-terminal configuration, however these measurements turned out to be inaccurate since the LCR-meter cannot properly measure samples with a high contact resistance between the current and voltage leads. Therefore the measurements yield inaccurate (higher than real) values for the capacitive part of the samples.

**D. Infrared Thermography Methodology**

IR thermography measurements are based on temperature increases due to Joule heating. The thermal radiation is measured and converted to temperature using an IR camera (FLIR ONE Gen 2 for Android, FLIR Systems) with a 160 x 120 pixels Lepton chip connected to a smartphone (Motorola Moto G3, Motorola Mobility LLC) using a dedicated app (FLIR ONE, FLIR Systems). The system is composed of a thermal camera and a visual camera of which the images are combined in the app. The set-up has a spatial resolution of approximately 3.5 pixels per mm. The measured radiation from the surface is strongly affected by: the heat generation, the design of the 3D-print (geometry and materials) and by the thermal environment (convection, conduction and radiation to the surroundings).
The temperature distribution determined from the measured radiation is compared qualitatively to the distribution from the simulated power dissipation density (the resistively dissipated power per unit volume). Actual heat transport may cause differences between simulations and measurements due to the indirect comparison (comparing power dissipation to measured thermal radiation). Furthermore the emissivity of the material is unknown, so the focus will only be on relative differences (which the low-cost camera can measure with sufficient accuracy [17]).

Several steps can be taken to have more control over the heat transfer. The samples are cooled actively at the bottom of the glass wafer using a fan to reduce thermal blurring by heat transfer. In case of dominant noise (e.g. from the surroundings) or small spatial temperature differences, lock-in thermography can also be applied. For this method the specimen is heated with an AC signal. The IR measurements are then analyzed in a pixel-wise manner at a frequency which is double the driving frequency [11] (or the driving frequency in case of a DC bias much larger than the AC driving signal). By analyzing the heating only at the relevant frequencies noise is removed. For the lock-in measurements the time-lapse mode from the FLIR app is used. A picture is made every 5 s (0.2 Hz) while the low frequency modulation is applied at 0.02 Hz.

Above the thermal cut-off frequency the sample response will not follow the electrical heating signal anymore. Furthermore the maximum sampling frequency of the FLIR camera is 9 Hz, giving a practical limitation to the height of the heating frequency. Amplitude modulation (AM) is proposed as solution for lock-in measurements at frequencies above the thermal cut-off frequency, which already has been applied successfully for vibro-thermography measurements of polymers and composites [18]. The high frequency electrical driving is modulated with a frequency well below the thermal cut-off and camera sampling frequency. The electrical currents are expected to follow the high frequency conduction paths while the sample temperature distribution can be measured at the modulated low frequency heating. For this set-up a signal generator with AM-functionality (HP33120A) is connected to a custom high-speed, high voltage amplifier (ESyLAB LM3326) to excite the samples. The average dissipated power can be calculated using:

$$P = \frac{1}{2} \Re\{UI^*\} = \frac{1}{2} \Re\left\{ \frac{U \cdot U^*}{Z} \right\}$$  \hspace{1cm} (1)

In this equation $U$ is the voltage in V, $I$ is the current in A, $*$ denotes the complex conjugated and $Z$ is the impedance in $\Omega$. Since at higher frequencies the capacitive contribution reduces the magnitude of the impedance $Z$, it is expected that at higher frequencies a lower voltage is required to obtain the same power dissipation. It has to be noted that by taking pictures the instantaneous radiation is measured (related to the instantaneous dissipated power, the averaging over the shutter time can be neglected because of the low modulation frequency), whereas in the simulations the average power dissipation density is calculated.

For small samples with a low capacitance and a high resistivity the difference in heating between low and high frequency is expected to be small. To show the value of AM with lock-in thermography a large capacitor sample is made with resistive electrodes. Two layers of ETPU of 100 mm by 10 mm and 0.2 mm thickness are printed on two polyimide films of 0.08 mm thickness. Subsequently, the polyimide films are stuck together back to back, functioning as the dielectric (with a thickness of 0.16 mm and relative permittivity of 3.5) of a capacitor. Crocodile clips are used to connect the amplifier to both electrodes. The heating pattern is then studied for amplitude modulation at various frequencies.

### E. Voltage Contrast SEM Methodology

The Voltage Contrast method makes use of the contrast that arises in SEM imaging upon a difference in electric potential. Electrons in an SEM at various potentials are decelerated or accelerated towards the detector in dependence of the potential of the sample surface. DC VCSEM measurements are done using an FEI Quanta 450, where the different leads have a different bias relative to the ground of the SEM. For the basics of SEM, readers are referred to [19]. A more positive bias reduces the number of secondary electrons reaching the detector, giving rise to a nonlinear dependency. Fig. 3 shows the theoretical signal versus bias of the low energy electrons, the so called secondary electrons (SE) [14]. An Everhart-Thornley detector is used, which uses a Faraday cage with 250 V bias to attract the low energy electrons. The SEM is used in low voltage operation, with an acceleration voltage of 1 kV. The low voltage operation increases the contrast (there are more low energy electrons that are sensitive to the local electric field) [14] and causes less sample degradation. The sample stage is placed completely at the bottom of the SEM, to have a full view of the samples (the maximum view is approximately 20 mm by 20 mm). The contrast and brightness are set automatically and the recorded images are inspected to make sure there is no clipping of the intensity over the full bias voltage range.

Several effects reduce image quality in SEM measurements. For example stochastic noise (shot noise) is part of the SEM signal and can be filtered best by median filtering [20]. Furthermore topography (edge effect) of the samples and
Fig. 3. The theoretical secondary electron (SE) signal intensity as a function of bias voltage. Positive voltages lead to a strong decrease of the number of SEs reaching the detector, while a negative voltage only slightly enhances the SE yield [14]. This curve is determined for every pixel of a sample, such that the inverse can be used to map measured signal intensity to voltages.

location of the detector (illumination effect) influence the measurements, together with the presence of micro and macro electric fields. To reduce these effects, a calibration curve is made using SEM images for which the sample is homogeneously biased by applying the same voltage (bias) to both leads. This results in a known potential everywhere in the sample and a sweep of these bias voltages is used to make a pixel-wise calibration curve to subsequently reconstruct the voltage distributions by normalizing, fitting and inverting the curves. Hence the curve in Fig. 3 is determined for every pixel separately and used to determine voltages from measured contrast.

For accurate quantitative VCSEM measurements a retarding field energy spectrometer is required [21]. Both micro and macro electric fields, e.g. from the sample and SEM chamber, influence the SE contrast formation. This results in different contrast at the same potential [14], [21] (although a large extraction field already gives reasonable results).

III. RESULTS

A. Modeling Results

The model results are shown alongside the experimental results. Both the simulated power dissipation density in Fig. 5 and the simulated voltage distribution in Fig. 12 are slightly skewed. This indicates a small amount of anisotropic conduction. Purely isotropic conduction would be indicated by a radially spreading power dissipation density or voltage from the connections in the corners.

B. Fabrication Results

The fabricated main parallel and opposite samples are shown in Fig. 4.a. A bit of roughness around the edges and traxel edges is present due to the printing process, influencing the physical behaviour and introducing an error in the fitted $\rho$ and $\sigma$. An initial roughness measurement on a section of the print with a white light interferometer (Polytec msa-500) indicated small ridges of 40 $\mu$m at the edges of traxels. In future samples this can be reduced by using ‘ironing’, a post-treatment method where the heated nozzle is moved over the printed layer without extruding material [22].

The capacitor AM sample is shown in Fig. 4.b, where the top electrode and polyimide film dielectric can be recognized. The AM sample is printed with two layers per side with the opposite infill angles (0$^\circ$ and 90$^\circ$ with respect to the length direction), to reduce the anisotropy effects.

C. Resistance Results

The total measured resistances are 67.4 k$\Omega$ for the sample with parallel leads and 60.7 k$\Omega$ for the sample with opposite leads. The fitted electrical parameters together with the geometrical parameters for the simulations are given in Table I. The visual fit to the voltage distribution is done for both samples with the same electrical parameters.

D. Infrared Thermography Results

The IR thermography results and simulations can be found in Fig. 5. The instantaneous thermal measurements in Fig. 5.c and 5.f give temperature distributions ranging from 20.5 °C to 43 °C. The simulations present peaks in power dissipation densities next to the leads (Fig. 5.a and 5.d) that do not
match the measured temperature distribution entirely. The high dissipation gradients will cause thermal conduction, yielding a more spread out temperature distribution. To illustrate this the plotted colour range of the average power dissipation value has been limited to a maximum value (Fig. 5.b and 5.e), showing a more comparable distribution to the measured temperature.

Fig. 6, top, shows the measured temperature intensity at three spots on the right sample from Fig. 4.a (hottest spot, sample edge and substrate), and at the bottom the corresponding power spectral density. The excitation signal had a bias of 25 V and an amplitude of 25 V. The pixel-wise filtering is done by taking out all signal except for the excitation frequency and its first harmonic (with a band of ±0.005 Hz around the excitation frequency peak and a band of ±0.01 Hz around the first harmonic peak). The conduction occurs directly between the two leads with some spreading through the rest of the sample. Furthermore, some conduction occurs through the edge opposite of the leads, caused by the smaller number of contacts between the bends as can be seen clearly in Fig. 7.b. This is something that cannot be seen in a DC measurement on the same sample, Fig. 5.f. From Fig. 7 it becomes clear that lock-in thermography can clear out the static heating and noise significantly. With this method it is not possible to precisely distinguish the power dissipation density between different traxels due to the diffusive thermal nature of the measurements. The merit of the method mainly lies in imaging the locations of power dissipation and the anisotropic conduction.

Amplitude Modulation was implemented for measurements above the thermal cut-off frequency, with the dedicated capacitor sample from Fig. 4.b. Significant heating could only be achieved for frequencies of 50 kHz and higher. The AM lock-in results are shown in Fig. 8 by means of the filtered, normalized intensities. For 50 kHz the signal is very small and just above the noise level, whereas for the higher frequencies the signal is more clear. For higher frequencies the heating is located more around the electrical connections, showing the use of lock-in AM for IR thermography. For future measurements more information on the thermal conduction can be obtained by also considering the phase information of the thermal measurements, which corrects for emissivity inhomogeneities [18], [23].
Fig. 9. An SEM image of a conductive, single-layer, 3D-printed sheet at an equipotential. From the “shadows” it becomes clear that the detector is placed at the top edge of the image. The edge and topographic effect clearly show the printed traxel geometry. The white bar on the lower right indicates 3 mm.

Fig. 10. Selection of pixel-wise calibration curves after median filtering without normalization. Depending on the location of the pixel the intensity is very high and maximum for negative bias voltages. Every curve represents the measured values corresponding to the theoretical calibration curve in Fig. 3.

E. Voltage Contrast SEM Results

In Fig. 9 a close-up of an SEM measurement of a 3D-printed sample at a constant voltage is shown. The effects due to the rough surface can be seen (the edges have a higher intensity) and the detector is positioned at the top of the image (giving rise to the shadows, characteristic to the illumination effect). Furthermore the noise can be recognised in smooth areas.

The effects are reduced by pixel-wise calibration, Fig. 10 shows measured calibration curves (median filtered) for a part of the pixels, where depending on the location and voltage the intensity can vary significantly. Some pixels show the characteristic shape from Fig. 3 whereas others have a maximum intensity lower than 1 which is not constant for negative bias voltages because of the complex effects occurring in the SEM.

Fig. 11 shows SEM images as used for pixel-wise calibration (11.a, 11.b, 11.d, 11.e) and as used for voltage distribution analysis (11.c, 11.f). The calibration images have the same grey-tone everywhere in the samples, apart from the topographic and edge effects of the SEM. The measured voltage distribution images show a clear transition in contrast between both leads. Fig. 12 gives the corresponding FEM simulations and experimental results, showing both qualitatively and quantitatively good correspondence. The results show a small amount of anisotropic conduction in the samples, since the voltage distribution extends more along the traxels than over the traxel’s interfaces. The effect is small due to a low contact resistance (Tab I). The spatial resolution of the SEM measurements is very high. The resolution of the measured voltage is currently limited by the (filtered) stochastic noise and the mapping of intensity to voltage with the calibration curves. The high spatial resolution of the method allows to measure the voltage of individual traxels accurately.

IV. SENSOR APPLICATIONS

To demonstrate the usefulness of IR thermography and VCSEM for characterization of flexible, sheet-like sensors; the methods have been applied to three sheet-like sensors with a suitable impedance. The focus lies on 3D-printed sensors...
because of their potential. However VCSEM and IR thermography can equally well be applied to other sheet-like resistive sensors, particular when the distributed electrical properties or the anisotropy of the conductors are of importance, and as long as the sheets allow geometrically for the respective observations. This would hold e.g. for sensors based on screen-printed films, sputtered films, inkjet printed films and carbon fiber sheets.

A. Meander

The previous samples display only little anisotropic conduction. To study a sensor geometry with primarily anisotropic conduction, a single meandering traxel is studied. The meander has been printed on a polyimide film covered glass wafer, where Fig. 13.a shows the CAD design and 13.b the realized print. This geometry resembles common strain sensors and has no significant traxel interface areas (there is some overlap between traxels at the bends only). The realized print spans approximately 10 mm by 10 mm by 0.2 mm, with a traxel width of 0.4 mm and gaps of 0.5 mm.

The VCSEM measurement (Fig. 14.a) clearly shows a change in intensity over the traxel length. Where the samples in Fig. 12 have an almost isotropic resistivity, the meander sample shows anisotropic conduction: from top to bottom the intensity changes significantly, whereas from left to right the intensity drop is much smaller. Physically this can be explained by the current fully following the traxel since the traxels are only connected to their neighbours at the outer left and right ends.

In case the traxel has the same cross-section everywhere, it should have the same power dissipation density everywhere. The instantaneous IR measurement shows a bright square with lower temperatures around the edges. Individual traxels cannot be recognised due to the thermal and/or optical blurring. For better measurements lock-in thermography should be applied with a higher excitation frequency. This reduces thermal blurring, increasing the spatial resolution at the cost of signal strength [23]. Anisotropy can be studied with IR thermography qualitatively by studying the spread of heating. For a pure meander heating is everywhere the same, since the cross-section is everywhere the same. For an isotropic sheet, heating mainly occurs in the corners with the electrical connectors due to the smaller cross-sections.

These measurements can also be performed on a strain sensor under operation. In this way it will be possible to measure the effect of the deformation on the change in voltage distribution and power dissipation or to locate the areas that affect the change in resistance the most. For a deformed sensor a new set of calibration curves has to be made for every deformed configuration with VCSEM, while for IR thermography the heat flux on the camera depends on the camera and sensor orientation.

B. Pencil-on-Paper Sheet

The 3D-printed samples suffer from topographic effects with VCSEM. For measurements on a smoother surface a pencil-on-paper sample is studied (a paper sheet with high resistance from a layer of distributed graphite particles), where also the homogeneity of the resistivity can be studied. In literature reliable pencil-on-paper sensors have been shown, e.g. strain gauges and chemiresistors [24], [25]. The sample is made by manually coloring a square piece of A4 printer paper (10 mm by 10 mm) with a soft dark pencil (soft and dark indicates high graphite and low clay content). The sample is placed on a polyimide film as dielectric and silver paint-copper pad connections are used (Fig. 15.a). A close-up of the pencil-on-paper sample is shown in Fig. 15.b with the cellulose fibers and graphite deposits. From this close-up it becomes
square connection pads (at the top in Fig. 17.a) and excitation frequency of 1 MHz is modulated with a frequency of 0.04 Hz. The narrow vertical parts between the connection pads and the broad horizontal bridge serve as the strain sensing parts. The instantaneous thermal image in Fig. 17.b shows the heating of the strain sensor in combination with noise and IR reflections. By means of the lock-in procedure the noise and reflections can be filtered, Fig. 17.c. The result shows that the current mainly follows the shortest path (the top edge of the horizontal bridge) and causes most of the heating in the narrow strain gauges (showing their effectiveness), whereas the connection pads show almost no heating. The center areas of the strain gauge actually become the hottest and don’t fully cool down during modulation whereas the edges do fully cool down. Static heating in the center of the strain gauge is filtered, yielding the darker center areas compared to the bright edges. This is a drawback of the method which can be tackled by more active cooling or lower modulation frequencies.

V. DISCUSSION AND CONCLUSIONS

Characterization methods have been presented to investigate the electrical properties of 3D-printed conductors in combination with a physical model for FEM simulations. Infrared thermography and voltage contrast SEM have been applied for characterization, showing promising results. The IR thermography method has to be improved to enable reliable quantitative measurements, e.g. by including a thermocouple on the sample to measure the absolute temperature as well as by determining the thermal emissivity of the ETPU. Lock-in thermography can be used to reduce the effects of static heating and noise, where for frequencies above the thermal cut-off frequency amplitude modulation can be applied. Furthermore the power dissipation simulations have to be extended to the thermal domain to be able to properly compare the results to the measurements. The cooling conditions also need to be better defined, to enable proper heat transfer simulation. The voltage contrast scanning electron microscopy method (VCSEM) produces qualitatively good and quantitatively reasonable results. The complexity of the physics and SEM make it difficult to obtain accurate quantitative results. Intensity and contrast of the samples in the SEM can change over time, affecting the measurements, possibly because of Joule heating, sample degradation and substrate and material charging. Therefore the method can still be improved and also extended to AC measurements. The VCSEM and IR measurements correspond well with each other, showing a small amount of anisotropic resistivity for the samples used. Furthermore VCSEM and IR thermography have been applied both to sheet-like samples to show the possibilities for sensor characterization. Information on the voltage distributions and anisotropy for both isotropic and anisotropic conduction could be provided. Furthermore the methods allowed pinpointing of inhomogeneous resistivity and areas of higher resistance. The measurement methods complement each other when applied both. Future work will focus on analytical modelling, the relation between the printer settings and electrical properties and on improving and extending the characterization methods.

C. 3D-Printed Flexible Tactile Sensor

Finally IR thermography is applied to a differential, flexible tactile sensor [26], Fig. 17.a, to locate the most resistive areas. The sensor consists of a non-conductive, semi-transparent TPU cantilever (22 mm by 30 mm by 2 mm) with embedded piezo resistive strain gauges made of 3 layers of conductive, carbon-black filled TPU (ETPU) with a total thickness of 0.6 mm. Crocodile clips are connected to the two printed
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