
Silicon field emitter arrays – High current devices for harsh environments and novel characterization methods

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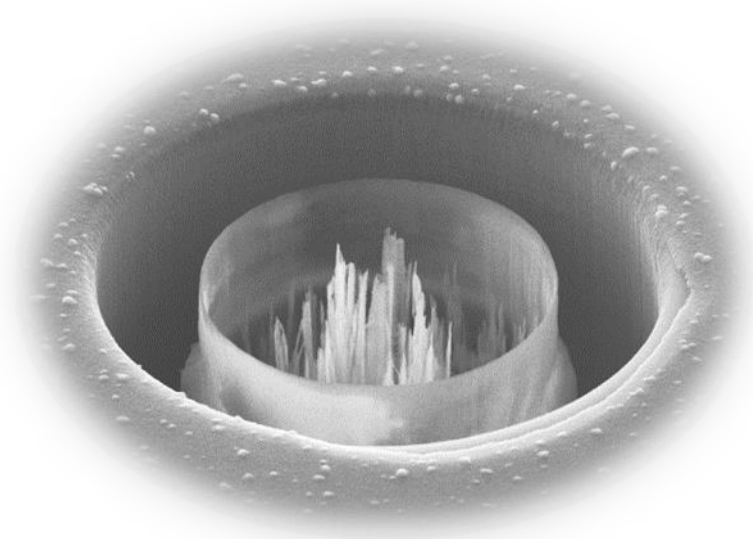
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DISSERTATION

Written by

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Declaration

I declare that this thesis is my work, and its contents were obtained and analyzed by myself, if not stated otherwise. This thesis is submitted to the Institute of Physics, EIT 2, Faculty of Electrical Engineering and Information Technology at the University of the Bundeswehr Munich and is not under consideration at any other German or foreign university.

Andreas Schels

Abstract

Field emitter arrays (FEAs) play a pivotal role in many vacuum electronic devices. In comparison to cathodes based on thermionic emission, they offer several advantages, including high-frequency switching capability, low power consumption, superior brightness and coherence, and a narrow energy distribution of the emitted electrons. These characteristics make FEAs promising candidates for high-current electron source applications, such as microwave power amplifiers or mobile fast-switching X-ray sources. Literature has shown the required current densities for these applications with various FEA designs under laboratory conditions. However, the commercial success of FEA-based products is still limited as ideal ultra-high vacuum environments typically differ from actual application conditions. This thesis presents a new FEA design for high-current and high-pressure operation based on silicon nanowires (SiNWs) with an integrated extraction gate.

First, the current dependent performance of ungated SiNW FEAs is compared to that of former anisotropically wet-etched pyramidal FEAs in diode configuration measurements. The SiNW FEAs exhibit a tenfold higher maximum current during current-voltage sweeps at similar applied electric fields. Emission currents of up to 2.5 mA can be maintained for one hour within the given extraction voltage limit of 1.4 kV at a base pressure of 10^{-5} mbar, even after preceding continuous operation of the sample for over 8 hours at various current levels at or above 100 μ A. A study on the influence of differently doped base materials reveals potential for homogenizing the current load distribution across the array by exploiting the local current emission limiting properties of p-type silicon FEAs.

The gated SiNW FEA design incorporates thermally oxidized vertical trenches between the emitting structures and the gate to minimize the risk of excessive leakage currents at high operating pressure levels. The electrical characterization of the devices in triode configuration confirms stable transmission rates of over 95% during constant current operation at 10^{-5} mbar, regardless of the emission current level. A maximum emission current of 630 μ A is demonstrated in continuous operation for one hour without noticeable degradation at a mostly constant extraction voltage of 280 V and a pressure of 10^{-5} mbar. This was achieved even after 24 hours of sample operation at various current levels during preceding lifetime testing.

Finally, a novel characterization method is introduced that measures the emission of individual emission sites in a spatially resolved and quantifiable manner during the integral operation of FEAs. First measurements reveal unprecedented insights into the doping dependent emission behavior of silicon FEAs. These insights can be used in future research to further optimize the lifetime of gated SiNW FEAs.

Kurzfassung

Im Vergleich zu thermischen Kathoden bieten Feldemitterarrays (FEAs) viele Vorteile, wie kurze Schaltzeiten, geringen Stromverbrauch sowie eine hervorragende Helligkeit und Kohärenz des Elektronenstrahls. Daher eignen sie sich als Hochstrom-Elektronenquellen, beispielsweise in Wanderfeldröhren oder mobilen, schnell schaltenden Röntgenquellen. In der Literatur wurden bereits mehrfach die für diese Anwendungen erforderlichen hohen Stromdichten unter Laborbedingungen im Ultrahochvakuum gezeigt. Der kommerzielle Erfolg von FEA-basierten Produkten ist jedoch nach wie vor begrenzt, da diese idealisierten Umgebungen häufig von den tatsächlichen Anwendungsbedingungen abweichen. In dieser Arbeit wird ein neues FEA-Design mit integriertem Extraktions-Gate vorgestellt, das auf Silizium-Nanodrähten (engl.: *silicon nanowires*, SiNWs) basiert und für die Emission hoher Ströme bei hohen Umgebungsdrücken optimiert ist.

Zunächst wird die Stromtragfähigkeit von SiNW FEAs ohne Gate mit der von pyramidalen FEAs in Diodenkonfigurationsmessungen verglichen. Die SiNW FEAs weisen während Strom-Spannungs-Sweeps einen zehnfach höheren Maximalstrom bei gleichen angelegten elektrischen Feldern auf. Emissionsströme von bis zu 2,5 mA können innerhalb des Extraktionsspannungslimits von 1,4 kV bei einem Basisdruck von 10^{-5} mbar über eine komplette Stunde hinweg aufrechterhalten werden. Dies gelingt selbst nach vorherigem Dauerbetrieb der Probe für über 8 Stunden bei verschiedenen Emissionsströmen von über 100 μ A. Eine Studie zum Einfluss unterschiedlich dotierter Basismaterialien zeigt Potenzial zur Homogenisierung der Stromlastverteilung über das Array durch gezielte Limitierung der lokalen Ladungsträgerversorgung mittels p-dotiertem Silizium.

Das neue Design der SiNW-FEAs mit integriertem Gate inkludiert thermisch oxidierte Gräben um die emittierenden Strukturen. Dadurch wird die Kriechstrecke zum Gate minimiert und das Risiko exzessiver Leckströme bei hohen Betriebsdrücken reduziert. Die elektrische Charakterisierung in Triodenkonfiguration zeigt eine stabile Transmissionsrate von über 95 % im Konstantstrombetrieb bei 10^{-5} mbar, unabhängig vom Emissionsstromlevel. Im einstündigen Dauerbetrieb bei einem Druck von 10^{-5} mbar wurde ein Emissionsstrom von 630 μ A bei einer Extraktionsspannung von 280 V ohne merkliche Degradation gezeigt.

Abschließend wird eine neu entwickelte Charakterisierungsmethode vorgestellt, die eine orts aufgelöste Messung der lokalen Teilemissionen während des integralen Betriebs von FEAs ermöglicht. Erste Messungen enthüllen bereits neue Einblicke in das dotierungsabhängige Emissionsverhalten von Silizium-FEAs. Die Methode kann somit in weiterführenden Arbeiten genutzt werden, um die Lebensdauer der gated SiNW FEAs weiter zu optimieren.

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Abbreviations

FE	Field emission
FEA	Field emitter array, field emission array
JWKB	Jeffrey-Wentzel–Kramers–Brillouin
MG	Murphy-Good
SN	Schottky-Nordheim
FEF	Field enhancement factor
IV	Current-voltage
ML	Millikan-Lauritsen
UHV	Ultra-high vacuum
CNT	Carbon nanotube
SiNW	Silicon nanowires
MOSFET	Metal-oxide-semiconductor field-effect transistor
TMAH	Tetramethylammonium hydroxide
RIE	Reactive ion etching
DRIE	Deep reactive ion etching
HF	High-frequency
ICP RIE	Inductively coupled plasma reactive ion etching
DAC	Digital-to-analog converter
ADC	Analog-to-digital converter
PEEK	Polyetheretherketone
OPAMP	Operational amplifier
CDLT	Current-dependent lifetime test
CCM	Constant current measurement
SEM	Scanning electron microscope
QFEI	Quantitative field emission imaging
DN	Digital number

1 Introduction

Electron sources play a pivotal role in numerous technological domains. The mechanism responsible for electron emission in such devices has a significant impact on the specific electron beam characteristics, which determine the ideal area of application. The basic types of classic electron sources can be divided in thermal emission, field emission (FE), photoelectron emission, and secondary emission cathodes. FE-based cathodes, in particular, offer many advantages compared to the widely used thermal emitters. Due to their ability to obtain free electrons without the need for external heat, these devices exhibit low power consumption, high frequency switching capabilities, and reduced thermal loads [1]–[4]. Furthermore, they are recognized for their superior brightness [5], [6], high coherence [6], [7], and narrow energy distribution of the emitted electrons [5]–[8]. This makes FE based electron sources promising candidates for a variety of applications, such as X-ray devices [1], [5], [9]–[13], microwave amplifiers [2], [14]–[16], terahertz laser diodes [17], mobile electron guns [18], and vacuum channel transistors [19], [20].

To meet the high-current requirements of many applications, field emitter arrays (FEAs) are commonly utilized. FEAs typically consist of numerous tips that emit in parallel, enabling the total current load to be distributed over multiple individual emitters. Recent advancements in nanotechnology have made it possible to realize millions of individual emitters on even the smallest device footprints. Although there have been numerous publications on various applications, there are only very few commercial products based on field emitters. One reason for this is, that the FEAs shown in the literature are usually operated under ideal conditions in an ultra-high vacuum, which provides the best performance. However, in many applications, it is often not possible to maintain such a low pressure. Pressure levels closer to the application can cause premature failure and significantly reduce the performance of FEAs. To achieve commercial success of FE-based applications, a reliable emitter with sufficient lifetime and emission current stability, even under harsh conditions, is necessary.

The long-term objective of our project is to develop a portable X-ray source based on field emission technology that can match the lifetime and current output of commercial products based on thermal emitters, while also providing the aforementioned benefits. To achieve this, the electron source must be able to supply emission currents in the range of 0.1 to 10 mA, ideally for at least 10,000 hours [13], [21]. The challenge in this case is that the pressure in handheld devices is significantly higher than the typical operating conditions of FEAs, as active pumping is not possible. The actual pressure level depends on the final sealing process

used for the product. However, vacuum-sealed devices typically operate at a pressure level of greater than 10^{-6} mbar [22], [23]. In some cases it may even be in the range of 10^{-5} mbar [18]. The goal of this thesis is to develop a robust FE electron source capable of delivering emission currents of at least 100 μA in constant current operation at a base pressure of 10^{-5} mbar. To achieve this, a comprehensive literature review of various FEA materials and concepts is conducted to reveal the current state-of-the-art field emitter arrays for high-current applications and establish the foundation for this work. Silicon is chosen as the substrate material for this work due to its similar performance to other base materials and the wide range of possibilities offered by semiconductor process tools. Various silicon FEAs with different emitter morphologies are developed and electrically characterized at the desired pressure range in diode configuration in a comparative manner. The most promising type is then incorporated into a novel FEA design with a fully integrated gate. The design aims to suppress leakage currents into the gate, even at high pressure levels, and deliver high transmission currents under harsh operational conditions. Furthermore, a new measurement method is introduced that detects emission currents from each individual emission site during the integral operation of the FEA. This provides unprecedented insights into the emission behavior of FEAs, which can be used for further optimization of the developed devices in future work.

2 Field Emission Theory

In both classical and quantum physics, emission of an electron from a solid object into a vacuum requires the electron to overcome a potential barrier. In classical physics, this necessitates a supply of energy, which may be derived from various sources such as thermal emission, photoelectron emission, or secondary electron emission from high-energy particles. Field emission, also known as field electron emission or cold field electron emission, provides a quantum mechanical option that does not require an external energy supply. In this process, tunneling is used to surpass the potential barrier. The development of the Schrödinger equation [24] and the Jeffrey-Wentzel–Kramers–Brillouin (JWKB) approximation [25], [26] established the groundwork for interpreting this phenomenon. *Fowler and Nordheim* provided the first conclusive description in 1928 [27], [28], although they made a small mathematical error [29] that was not corrected until 1953 by *Burges, Kroemer and Houston* [30]. The theory was later extended by *Murphy and Good* in 1956 [31] by incorporating a more realistic tunneling barrier and factoring in thermoionic emission at non-zero temperatures. Additional extensions and mathematical approximations subsequently culminated in the proposal of a universal formulation for field electron emission by *Forbes et al.* [32], which is based on the 1956 Murphy-Good (MG) field emission theory. Nonetheless, a comprehensive theoretical model remains a topic of ongoing research (see Chapter 3.1). All evaluations performed in the scope of this thesis, rely on the *extended Murphy-Good equation* [33], [34]. Therefore, this chapter concisely explains the fundamentals of this theory and its applicability in the analysis of field emission data. An informative overview of the history of and theory behind various other existing models can be found for example in Chapter 9 of [21].

2.1 Field Emission from Smooth Planar Ideal Metals

We are looking for a formula that describes the field emission current as a function of the electric field applied to the emitting structure. To do this, we start with a Sommerfeld-type free-electron model of an ideally smooth planar metal-like emitter. Figure 2.1 shows the one-dimensional potential barrier at the metal-vacuum interface of such a material. At a temperature of 0 K, all energetic states below the Fermi energy E_F are occupied. The height of the potential barrier preventing the electrons from leaving the solid is defined by the work function Φ , which equals the difference between the vacuum potential and the Fermi energy.

In the absence of external fields, the shape of this potential barrier can, to a first approximation, be described as a step function. When an external electric field is applied, it forms approximately a triangular barrier.

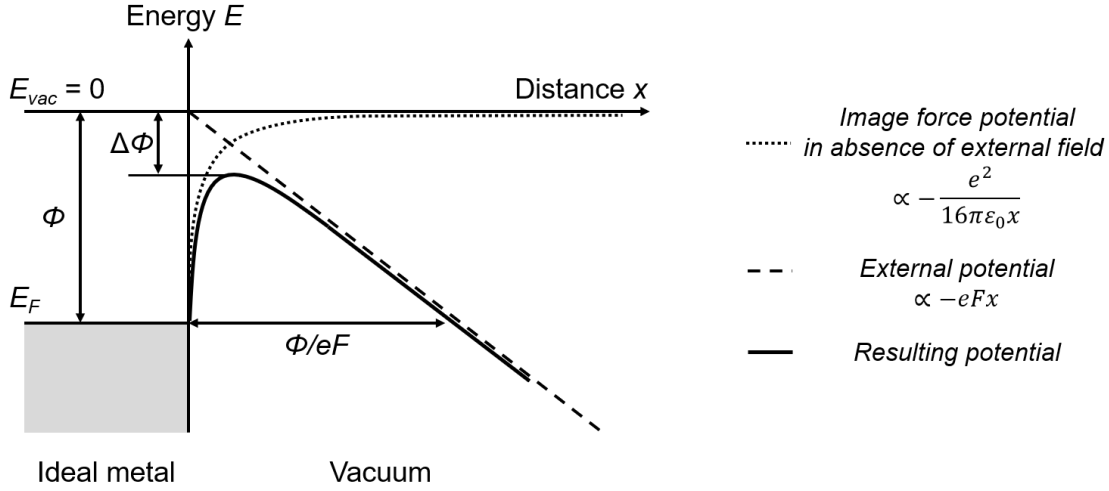


Figure 2.1 Band diagram for an ideal Sommerfeld-type metal in vacuum.

It is known from electrostatics that an emitted electron at a finite distance from the surface of an ideal conductor is attracted by the so-called image force potential (dotted line in Figure 2.1) [35]. This reduces the height of the resulting barrier (solid line) by $\Delta\Phi$. In total, potential barrier seen by an emitting electron ($x > 0$) when a constant external field is applied can thus be written as

$$E(x) = E_F + \Phi - eFx - \frac{e^2}{16\pi\epsilon_0 x}, \quad (2.1)$$

where F is the electric field strength, e is the electron charge and ϵ_0 is the vacuum permittivity. This form of potential barrier is now commonly known as the Schottky-Nordheim (SN) barrier [21]. The maximum of this function describes the reduction $\Delta\Phi$ of the effective barrier height by

$$\Delta\Phi = \sqrt{\frac{e^3 F}{4\pi\epsilon_0}}. \quad (2.2)$$

By applying a sufficiently strong electric field to the metal surface, the width of the resulting barrier can be brought into the range of the de Broglie wavelength of electrons. This results in a finite tunneling probability, here represented by the transmission coefficient $T(E, F)$. Together with the number of electrons impinging on the surface $N(E)$, the emitted current density can be calculated as [36]

$$j(F) = e \int_0^\infty T(E, F) N(E) dE . \quad (2.3)$$

Due to the assumption of a Sommerfeld-type electron gas, $N(E)$ can be determined from the Fermi-Dirac distribution [21]. The transmission coefficient requires the solution of the Schrödinger equation, which cannot be done analytically for the SN barrier. By applying the JWKB approximation, an equation for the emission current density is obtained, which is known as the Murphy-Good equation [31]. For detailed descriptions of the mathematical procedure, see for example Chapter 1 of [36] or Chapter 8 and Chapter 9 of [21]. The resulting equation can be written as

$$j(F) = \frac{A_{FN}}{\Phi t(f)^2} F^2 \cdot \exp\left(-\frac{B_{FN} \Phi^{3/2} v(f)}{F}\right), \quad (2.4)$$

where A_{FN} and B_{FN} are known as the first and second Fowler-Nordheim constants, with $A_{FN} = e^3/(8\pi h) \approx 1.5414 \cdot 10^{-6} \text{ A} \cdot \text{eV} \cdot \text{V}^2$ and $B_{FN} = 8\pi(2m_e)^{1/2}/(3he) \approx 6.8309 \cdot 10^9 \text{ eV}^{-3/2} \cdot \text{V/m}$ [34]. The special mathematical functions $t(f)$ and $v(f)$ account for the SN barrier and do not have an analytical solution. However, tabulated solutions obtained by various methods can be found in the literature [37], [38]. Alternatively, the following approximations published by *Forbes and Dean* [38], [39] can be used, resulting in minor relative deviations of 0.33% or less [40]:

$$v(f) \approx 1 - f + \frac{1}{6} f \ln(f) \quad (2.5)$$

$$t(f) \approx 1 + \frac{1}{9} \left(f - \frac{f}{2} \ln(f) \right) \quad (2.6)$$

The herein used variable f is called the so-called scaled barrier field, which is defined via the reference field F_Φ at which the height of this SN barrier is reduced from Φ to zero. Based on Equation (2.2) and the Schottky constant $c_S = (e^3/4\pi\epsilon_0)^{1/2} \approx 3.7947 \cdot 10^{-5} \text{ eV} \cdot (\text{V/m})^{-1/2}$ [41] it is defined by

$$f = \frac{F}{F_\Phi} = \frac{c_S^2}{\Phi^2} F . \quad (2.7)$$

As a function of the applied field, it describes the shape of the tunneling barrier and thus the emission regime in which an emitter is operated [42]. It can be used to evaluate the applicability and validity of certain approximations and models [43]–[45].

Note that, inserting the approximation from Equation (2.6) into Equation (2.4) leads to the fact that the formula can no longer be linearized, which is a useful property for empirical data analysis (see Chapter 2.4). Forbes [33], [46] suggests incorporating $t(f)$ into an uncertainty pre-factor λ “of unknown functional behavior” [33] that takes into account all the effects of the approximations in this model. Deviating from this, the approximation $t(f)^2 = 1.1$ is used in this work, which is also common in other literature [47], [48].

Based on these approximations, it is now possible to describe the emission current as a function of the electric field by integrating over an effective emission area A_{eff} . For better readability, two new parameters κ and η are introduced, which correspond to $\kappa = 2 - \eta/6$ and $\eta = B_{FN} c_s^2 \Phi^{-1/2} \approx 9.836239 \text{ (eV}/\Phi)^{1/2}$. The resulting formula looks as follows

$$I(F) = A_{eff} \frac{A_{FN} \Phi^{3-2\kappa}}{c_s^{4-2\kappa} 1.1} F^\kappa \cdot \exp\left(\frac{B_{FN} c_s^2}{\Phi^{1/2}} - \frac{B_{FN} \Phi^{3/2}}{F}\right). \quad (2.8)$$

2.2 Field Enhancement for Non-Ideal Emitter Surfaces

Up to this point we treated our ideal metal-like emitter as a perfectly smooth planar surface of macroscopic expansion. The necessary electric field strengths required to extract measurable currents from such surfaces are typically in the range of several V/nm [21], [49], [50], which is unattractive for technical applications. However, it is well known from electrostatics that the electric field inside an ideal conductor is always zero. Therefore, when a conductive protrusion (e.g. surface roughness) is placed in a homogeneous electric field, it will compress the equipotential lines and thus increase the electric field at its highest point. This phenomenon is called field enhancement and is expressed by the field enhancement factor (FEF) γ . Because of its importance for understanding field emission, the geometric dependencies of the FEF have been studied and modelled extensively in the literature. Researchers came to an understanding, that for an ideal Sommerfeld-type system the FEF solely depends on the emitters geometry [51]. A very detailed and up-to-date review on this subject is provided by *de Assis et al.* [52]. Almost all models in the literature agree that the FEF is highly dependent on the height and radius of curvature of the protrusion. In a first approximation, it can even be calculated from the height-to-radius ratio [51], [53]. Therefore, tip-like or post-like geometries are commonly used in field emitters to locally increase the extraction field by several orders of magnitude [51]. In addition to the radius of curvature and the height, the distance between neighboring emitters in FEAs, the distance to the collector and their ratio also affect the FEF [54]. Steeper angles can also increase the FEF, while non-geometric

effects such as vacuum space charge and internal series resistance in non-ideal conductors can decrease it [52].

To account for these effects when predicting the emission current of a field emitter, γ must be added to Equation (2.8) as a pre-factor to the electric field F . In addition, the field is replaced by the voltage V according to $F = V/d$ with the aid of the tip-to-anode distance d , since measurements are usually voltage related. This gives the final form of the field emission equation used in this thesis

$$I(V) = A_{eff} \frac{A_{FN} \Phi^{3-2\kappa}}{c_S^{4-2\kappa} 1.1} \left(\frac{\gamma V}{d} \right)^\kappa \cdot \exp \left(\frac{B_{FN} c_S^2}{\Phi^{1/2}} - \frac{B_{FN} \Phi^{3/2} d}{\gamma V} \right). \quad (2.9)$$

It describes the field emission current from a perfect conductor assuming a uniform and constant local work function and a planar emission surface. It is important to keep these approximations in mind, as they cannot be satisfied by real field emitters. The work function is not constant for real emitters, since current-induced Joule and/or Nottingham heating [55], [56] and associated changes in adsorbate coverage can affect it [44] (see Chapter 3). The planar assumption is only applicable for “larger” radii of curvature > 20 nm [57], [58], since it affects the local electron supply to the inside of the emitter surface and the transmission probability. Furthermore, V is not necessarily the emission voltage, in case of series resistances in the system or emitter, possible leakage currents and vacuum space charge. Nevertheless, this kind of approach is common practice in literature, due to the lack of a better theory. The consequences resulting from this are further discussed in Chapter 3.1.

2.3 Field Emission from Semiconductors

To date, there is no all-encompassing theory that can consistently describe field emission from semiconductors [21], [36], [49]. A good overview of the existing theories can be found for example in Chapter 5 of [59]. Here, we are going to focus on the relevant parts for the observations and assumptions made in this thesis. The decisive difference in the emission behavior of semiconductors, as opposed to ideal conductors or metals, is the density of charge carriers in the interior. For example, the concentration of silicon at room temperature is around 10^{10} cm^{-3} [60], whereas for metals it is in the range of 10^{23} cm^{-3} [61]. Due to the associated electric field penetration, semiconductors, unlike metals, have a relative permittivity ϵ_r unequal to 1. This in turn affects the image charge correction of the SN barrier, so the scaled barrier field can be adjusted to [62], [63]

$$f = \left(\frac{\epsilon_r - 1}{\epsilon_r + 1} \right) \frac{c_s^2}{\phi^2} F. \quad (2.10)$$

In the case of silicon, with a dielectric constant of 11.7 [64], this gives a prefactor of 0.845. It has been shown, that the relative permittivity of semiconductors increases with the doping concentration [65], bringing this factor closer to 1. Additionally, the field penetration in semiconductors leads to a change of the charge carrier concentration near the surface, resulting in the formation of a space charge zone and a bending of the conduction and valence band [66]. This process is schematically represented in the band diagram of an n-type and p-type semiconductor surface in the presence of a high electric field, shown in Figure 2.2.

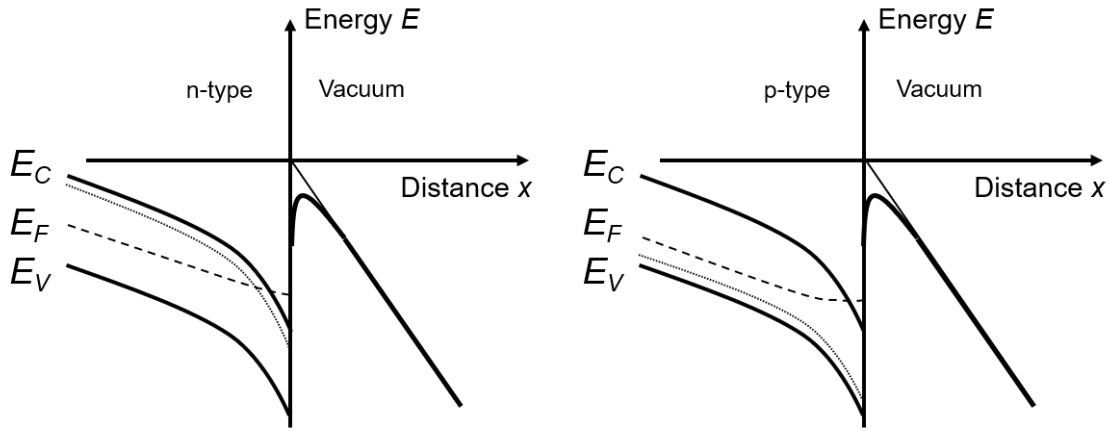


Figure 2.2 Energy diagram of the near-surface region a semiconductor in a high electric field for an n-type and p-type semiconductor. Adapted and reproduced from [63].

In the case of n-doping, this bending is less pronounced because it is counteracted by the surface states [49], [67]. In both cases, however, a sufficiently strong electric field will cause the conduction band to drop below the Fermi level, as shown in Figure 2.2. This leads to an accumulation of electrons near the surface, causing the now degenerate semiconductor to exhibit metal-like electronic behavior [68]. Since very high fields are usually present in field emission measurements, the addition to Equation (2.9) described in Equation (2.10) is neglected. The different barrier height for field emission in semiconductors, on the other hand, must be considered. Due to the dominance of conduction band emission, especially at high fields, the electron affinity should be used instead of the work function [69], [70]. For this reason, in all evaluations based on Equation (2.9) in this thesis, the work function is assumed to have a constant value of 4.05 eV, corresponding to the electron affinity of silicon [71].

Another non-neglectable effect in field emission from semiconductors is demonstrated in Figure 2.3. It shows the three stages of field emission behavior, that can be observed for p-

type and lightly doped (high resistive) n-type semiconductors materials. A very detailed explanation of this is given by *Liu et al.* in [70].

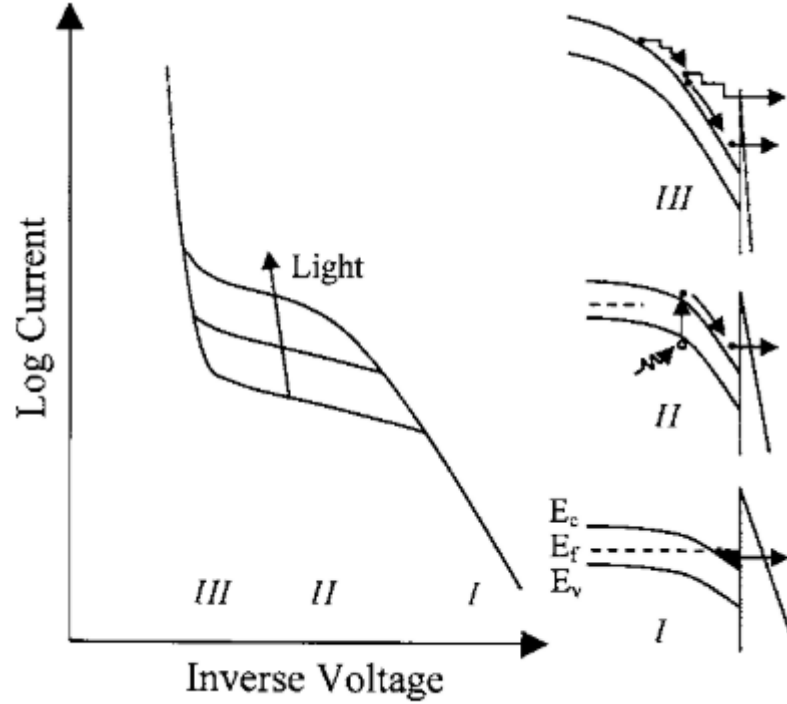


Figure 2.3 Illustration showing the energy-band structures and the field emission current characteristics for p-type semiconductors. Three regions have been experimentally revealed: (I) field-emission region limited by the tunneling probability of electrons, (II) depletion region limited by the supply of electrons to the conduction band at the surface, and (III) avalanche gain at very high electric fields in the space-charge layer. Reproduced with permission from [70]. Copyright 2006, Journal of Applied Physics.

As mentioned above, the typically high fields required for field emission can lead to an accumulation of electrons near the emitter surface. In a p-type semiconductor this is called an inversion layer. When the emission starts at relatively low voltages (Section I in Figure 2.2), the electron supply in the conduction band from this inversion layer is sufficient, so the emission current is mainly dominated by the tunneling probability of the electrons. *Stratton* [62], [72] proposed an analytical equation to describe this field emission current from the conduction band of a degenerate inversion layer. However, at low temperatures the characteristic can be approximated similarly to that of a metallic emitter [70], [73]. At increasing fields (Section II in Figure 2.2), the inversion layer begins to deplete until the emission current is strongly limited by the supply of electrons diffusing through or being generated in the depletion region. In an IV measurement this shows in the form of a saturation curve of the emission current. Due to the strong fields in the depletion region, the height of the saturation level is very sensitive to charge carrier generation, e.g. by thermal or photoexcitation [70], [74], [75]. The limitation due to the charge carrier supply also means that the emission current in this

region is relatively insensitive to changes in the tunnel barrier. As a result, possible changes in the work function due to adsorbates have less influence here, which means that a more stable emission current can be observed when p-type field emitters are operated in saturation [75]. If the extraction voltage exceeds a certain threshold, impact ionization can occur within the space charge region, which then leads to an avalanche effect and thus a strongly increasing emission current (Section III in Figure 2.2).

2.4 Analysis of Field Emission Data

In Equation (2.9) there are two main parameters that define the shape of the characteristic curve: the field enhancement factor γ and the effective emission area A_{eff} . A convenient method to extract these parameters from a current-voltage (IV) measurement using simple linear regression is by linearizing the field emission equation. For this purpose, the so-called FN plot was introduced in 1929 by *Stern et. al* [76]. Note, that this was based on the physically inadequate elementary emission equation of *Fowler and Nordheim* [27], [28]. According to the more accurate MG theory, which is also the basis for the analysis used in this work, this representation leads to a slightly curved line [29], [33]. Therefore, *Forbes* [33] proposed in 2019 to linearize the measurement data by plotting them in Murphy-Good coordinates. To do this, Equation (2.9) is transformed by dividing by the voltage and applying the natural logarithm to

$$\ln\left(\frac{I_m}{V_m^\kappa}\right) = \ln\left(A_{\text{eff}} \frac{A_{\text{FN}} \Phi^{3-2\kappa} \gamma^\kappa}{c_s^{4-2\kappa} 1.1 d^\kappa}\right) + \frac{B_{\text{FN}} c_s^2}{\Phi^{1/2}} - \frac{B_{\text{FN}} \Phi^{\frac{3}{2}} d}{\gamma} \cdot \frac{1}{V_m}. \quad (2.11)$$

The subscript “m” indicates the application to measurement data for I_m and V_m . This representation shows that plotting the measurement data on a coordinate system with $1/V_m$ as the x-axis and $\ln(I_m/V_m^\kappa)$ on the y-axis predicts an exactly linear course, assuming A_{eff} , γ and Φ as constants. Thus, by linear regression of the measured data in this coordinate system, the FEF can be calculated from the slope S_{fit} of the fit, according to

$$\gamma = \frac{-B_{\text{FN}} \Phi^{\frac{3}{2}} d}{S_{\text{fit}}}. \quad (2.12)$$

The effective area is then calculated from the y-axis intercept R_{fit} and the previously determined FEF γ by

$$A_{\text{eff}} = \left(R_{\text{fit}} - \exp\left(\frac{B_{FN} c_S^2}{\phi^{\frac{1}{2}}}\right) \right) \left(\frac{A_{FN} \Phi^{3-2\kappa} \gamma^{\kappa}}{c_S^{4-2\kappa} 1.1 d^{\kappa}} \right)^{-1}. \quad (2.13)$$

In this work, data sets are typically fitted in an automated manner using Python code. To avoid the misinterpretation of false data resulting from poor fits, the goodness of the fit is always checked. For this purpose, we calculate a chi-square value to evaluate the fit of the straight line to datasets with uncertainties in two coordinates. This is done as suggested in Chapter 15.3 of [77] using the formula

$$\chi^2 = \sum_{i=0}^{N-1} \frac{(I_{m,i} - S_{\text{fit}} - R_{\text{fit}} V_{m,i})^2}{(\sigma_{I,i}^2 + S_{\text{fit}}^2 \sigma_{V,i}^2) * (N-2)}. \quad (2.14)$$

Here, N represents the number of data points and σ_{yi} and σ_{xi} the uncertainties of the measured current and voltage, respectively. Large values of χ^2 generally indicate significant differences between the fitted and measured data, suggesting a poor fit, while small values suggest the uncertainties were possibly overestimated [77]. The tolerance limits utilized in this work were determined from empirical analyses performed on recorded data sets. Therefore, a fit is considered "good" if $\chi^2 < 20$.

Apart from the extraction of these emitter-specific parameters, the linearization of the measurement data also offers the possibility to check their validity. There are numerous possible errors that can occur when recording IV data (see Chapter 3.1). For example, due to the high voltages and electric fields, leakage currents can occur in the measurement setup, which can then be erroneously interpreted as part of the emission current. Since these rarely follow the regularity of tunnel current modelled in the equations above, they do not show linear behavior when plotted in MG coordinates. As a first check on the validity of FE data, it is therefore always advisable to plot the measured data in a linearized representation [44]. However, interpreting MG and FN plots is challenging due to their cryptic coordinate axes. A more easily accessible option is the Millikan-Lauritsen (ML) plot [78], which utilizes a semi-logarithmic form to plot the logarithmic emission current against the inverse extraction voltage. Similar to FN plots, ideal FE data represented in this form displays near-linear behavior [44], [46]. For this reason, a modified version of the ML plot, described in more detail in Chapter 4.2.3, is primarily used for data presentation and validity testing in this work.

3 Field Emitter Arrays – State of the Art

The evaluation and classification of publications on field emitter arrays is a difficult task, since the combination of theory and measurement, especially in the case of multipip field emission, is by no means trivial and still gives rise to controversial discussions [29], [44], [79], [80]. The exact measurement conditions and the way in which the data are interpreted are crucial for the quantitative results, which is why no reliably comparable figures of merit have yet emerged. The literature often examines macroscopic IV characteristics to determine microscopic area-like parameters or the superimposed field enhancement factor of the entire system [42], [51], [81]. Alternatively, maximum emission currents or current densities in relation to the emitter footprint are often specified. However, in both cases, it is important to consider the specific circumstances of data acquisition, which unfortunately are often insufficiently explained. Therefore, this chapter will first clarify the essential considerations for objectively comparing field emission data. Subsequently, a literature review of most widely used field emitter array concepts is provided to allow for classification of the emitters developed in this work. Note that this overview is based on the author's best knowledge, with no claims to completeness.

3.1 Comparability of Field Emission Measurements

The comparability of field emission measurements depends on two categories of factors: data acquisition factors (measurement conditions) and data evaluation factors (theory). Regarding data acquisition, the pressure in the measurement chamber has a significant impact on the emitter's performance, as it determines the density of residual gas particles available and, consequently, the supply of adsorbates. Contamination of the emitter's surface with adsorbates can affect the work function of the emitter and, thus, the transmission probability of the electrons [82]–[84]. *Spindt et al.* observed that changing the pressure from 10^{-9} mbar to 10^{-5} mbar can lead to a drop in the emission current of molybdenum FEAs by a factor of 10 due to oxygen poisoning [48], [85], [86]. However, the original emission could be restored by returning to lower pressures [85], [86]. In contrast, another measurement showed that hydrogen injection can increase the emission current by a factor of 20, again reversibly [85]. The energy required for the reversibility of these processes depends on whether the adsorbates are bound to the surface by van der Waals forces or through covalent bonding [84], [87]. In contrast to *Spindt*, where the adsorbates could be desorbed by the internally generated energy

from Joule heating, *Edler et al.* had to provide external energy by heating their silicon FEA to 110 °C to fully regenerate the degradation of the characteristic resulting from operation at an elevated pressure level of 10^{-3} mbar [83]. Therefore, it is evident that the emitter temperature also significantly affects the influence of surface adsorption. Recently, *Bhattacharya et al.* reported that the severity of silicon FEA degradation is not only affected by the pressure, but also by the gas composition [88], [89]. The water vapor content in the gas was found to be the primary factor contributing to an increase in work function. Additionally, it was demonstrated that a partial pressure of $5 \cdot 10^{-4}$ Torr argon causes greater degradation than the same partial pressure of nitrogen at a similar water vapor content. Notably, all degradations could be reversed by adsorbate removal, accomplished in this case through UV illumination. Conclusively, a complete absence of residual gas during the measurement would be necessary to ensure perfect comparability of field emission data. Besides the fact that this is technically unfeasible, a prediction of the emission behavior under actual application environments, which are frequently far from ultra-high vacuum (UHV) conditions, would become challenging in this case [18]. For this reason, it is essential to always consider the operating pressure during measurement when categorizing field emission data. In addition to this, potential pretreatment of the emitter can influence the emission characteristics. *Spindt et al.* observed an increase in maximum emission current of five to six orders of magnitude after pretreating their FEAs by heating under high electric field stress [85]. Also surface pretreatment by self-heating [90], [91], glow discharge before operation [92], external heating [93] and careful first turn-on [21], or UV exposure [94] can significantly improve the lifetime, stability and maximum emission currents of field emitters. Additionally, the type of FEA operation must be considered. Pulsed operation of field emitters is very common, but must be interpreted differently than constant dc emission, as the duty cycle has a decisive influence on the emitter's degradation, due to the different total current load over time. In connection with this, It has been demonstrated that field emission in microwave fields can decrease ion bombardment, a major factor in the irreversible degradation of emitters, by a factor of 10^6 [95]. Finally, the measurement setup itself must be considered, as different measurement results can occur depending on the configuration. Especially the distance between anode and FEA, as well as the shape of the anode can affect the data [54], [96], [97]. The design and layout of the emitter itself can also influence the results, especially with multi-tip field emitters [98].

Regarding data evaluation, the underlying theory plays the most decisive role. The mistake that can be avoided, but is still prevalent in the literature, is to use outdated and inaccurate theories as the basis for data evaluation [29], [79], [80]. However, even the best-developed and broadly accepted theory for field emission models the emitter as an ideal FE system in

the shape of a smooth, planar metal surface. Unfortunately, real emitters are commonly far from such ideal FE systems, especially since the rise of nanotechnology and their numerous materials and special morphologies [44], [80]. Possible reasons for deviations from the ideal include non-constant work functions due to temperature effects and adsorbate contamination, operation of the emitter in a regime outside of the Murphy-Good theory, or atomically sharp emitter tip radii, just to name a few [44]. Over the past few years, numerous theorists have made attempts to adjust the formula analytically to mitigate these deviations. These attempts include adaptations for nanometric radii tip curvature [57], [58], or for thermal field emission in general [99] and for the special case of nanowires [100]. However, discovering a comprehensive theory that integrates these partial aspects to enable flawless predictions of real experiments remains an ongoing area of research. To make matters worse, even if the theory utilized were entirely accurate, there remains the issue of measurement uncertainties. *Ayari et al.* found that even under UHV conditions at a base pressure of $4 \cdot 10^{-10}$ mbar and assuming a correct and constant local work function for the measured single tip tungsten emitter, the current noise leads to an uncertainty of up to 25% for the emission area and close to 1% for the FEF [101]. By including the uncertainties of the model, the fit and the work function, they conclude that an uncertainty of about 50% in the extraction of the FEF and the effective emission area is reasonable under good experimental conditions [80], [101].

In the case of field emitter arrays, the measured characteristic is typically a superposition of many tips emitting simultaneously, each with slightly varying morphologies and/or surface conditions. This results in multiple differing FEFs and overlapping partial emissions [102], [103]. Additionally, this thesis focuses on the development of FEAs suitable for usage in adverse environments, which require measurements under intentionally unfavorable experimental conditions that are far away from UHV. It is therefore to be expected that the theory can only be applied here to a very limited extent. Hence, the FEF and the effective emission area are analyzed and interpreted only in a subordinate manner in this work. Consequently, the focus of the presented studies and the following review of the state of the art is on the maximum current levels and densities that can be achieved by the FEAs, as these are at least not subject to data evaluation challenges. Nevertheless, as mentioned above, all figures of merit should always be interpreted in the light of the measurement conditions.

3.2 High-Current Field Emitter Arrays

The range of materials and morphologies of field emitters found in the literature is vast. Equation (2.9) shows that especially materials with low work functions or geometric structures with high FEFs are expected to have a high emission current. In many applications, field

emitters are expected to operate under adverse ambient conditions. Therefore, high chemical and mechanical stability, as well as high thermal and electrical conductivity, are advantageous. Especially in high-current applications, such as microwave power amplifier tubes or X-ray sources, multitip emitters like FEAs are often implemented [12], [104]. The following section introduces and reviews the most common types of FEAs used for this purpose.

3.2.1 Spindt Cathodes

Probably the best known and most extensively researched FEAs are the so-called Spindt cathodes or Spindt-type field emitter arrays. They are named after their developer, who published the first detailed description and characterization of these cathodes in 1968 [105]. They consist of an array of microfabricated conical molybdenum tips, each located in a cylindrical cavity of insulating thin film. Typically, the dimensions of the tips are in the micron or sub-micron range, with the number of tips and packing density depending on the application. On top of the insulating thin film there is a metallic counter-electrode, called a “gate”, which forms self-aligning electrode apertures around each tip. This type of FEA thus belongs to the FEAs with integrated gate, which will be discussed in Chapter 6. The gate is used in electron sources to apply the electric field required for extracting electrons from the microscopic tips, and the potential of the gate determines the emission current. In most applications free electrons are required. Therefore, a second electrode with a higher potential is commonly placed in some distance to the FEA, in order to transmit the electrons through the gate’s apertures. Thus, measurements of FEAs with integrated gate require three different potentials, one for the tips (typically ground), one for the extraction gate (typically between a few volts and up to kilovolts) and one for the anode (typically kilovolts). This measurement type is called a measurement in triode configuration, unlike measurements in diode configuration, where only two potentials are applied (to the tips and the extractor).

Over the years, Spindt-type FEAs have been tested in almost every standard field emission applications such as microwave amplifiers [2], [106], X-ray sources [107] or field emission displays [108], as well as in very specific applications such as mercury ion clocks for time measurement standardization [109]. *Schwoebel et al.* reported the highest emission current of such an FEA to date, with 330 mA at a pulsed extraction voltage of 160 V with 30 Hz pulses of 100 μ s duration from an array of 50,000 tips with a total area of 0.75 mm² [110]. This corresponds to a maximum emission current density of 40 A/cm². However, considering the duty cycle, a mean current density of 13.2 mA/cm² can be calculated for this based on the pulse duration and frequency. The pressure during the measurement was less than $2 \cdot 10^{-10}$ mbar and the emitter was pretreated by pulsed operation at 1.5 mA with 100 μ s pulses

at 30 Hz. The highest current ever measured from a single tip was 850 μA at 205 V, observed during dc sweep operation at a pressure of $2 \cdot 10^{-10}$ mbar by *van Veen* [111]. In this case the emitter was pretreated with a bakeout at 450 °C for 12 h, followed by a hydrogen sputter cleaning [111]. The highest current density of 1000 A/cm² was reported for a 16-tip array during half-wave rectified 60 Hz operation at 212 V. The FEA emitted a total current of 1.45 mA from an area of 0.0132 mm². The FEA was pretreated with a 450 °C bakeout. The measurement was performed under UHV conditions, which are not further explained in the publication [85].

3.2.2 Carbon-Based and Low-Dimensional Materials

Apart from these very well-known Spindt-type FEAs, there has been much interest in recent years in carbon-based [21], [112], [113] and/or low-dimensional materials [114], [115]. Many of those already found applications for example in microwave power amplifiers and X-Ray sources [12], [115]. However, due to the large number of existing publications and the high frequency of new publications in this field, it is hardly possible to make a comprehensive and complete comparison between all of them. Moreover, this would go beyond the scope of this work. Good reviews of many of these materials for field emission can be found for example in [13], [113], [116] or Chapter 10 of [21].

A very well researched overview of a variety of materials is given in Figure 3.1, which was reproduced from [13]. It shows the maximum current density (blue) together with the turn-on (green) and threshold electric field (red) for many FE cathodes to be found in the literature. The materials are arranged from left to right in increasing dimensionality. In addition, the work function increases from the left to the right within the same dimensionality. Each data point is usually based on at least three studies, taking into account only the material and not the morphology of the emitter. In order to compare the electric fields, the maximum current density was determined for each material. All current densities were then normalized to this value. The turn-on and threshold electric fields were then defined as the fields required to obtain 10% and 30% of the normalized maximum current density, respectively.

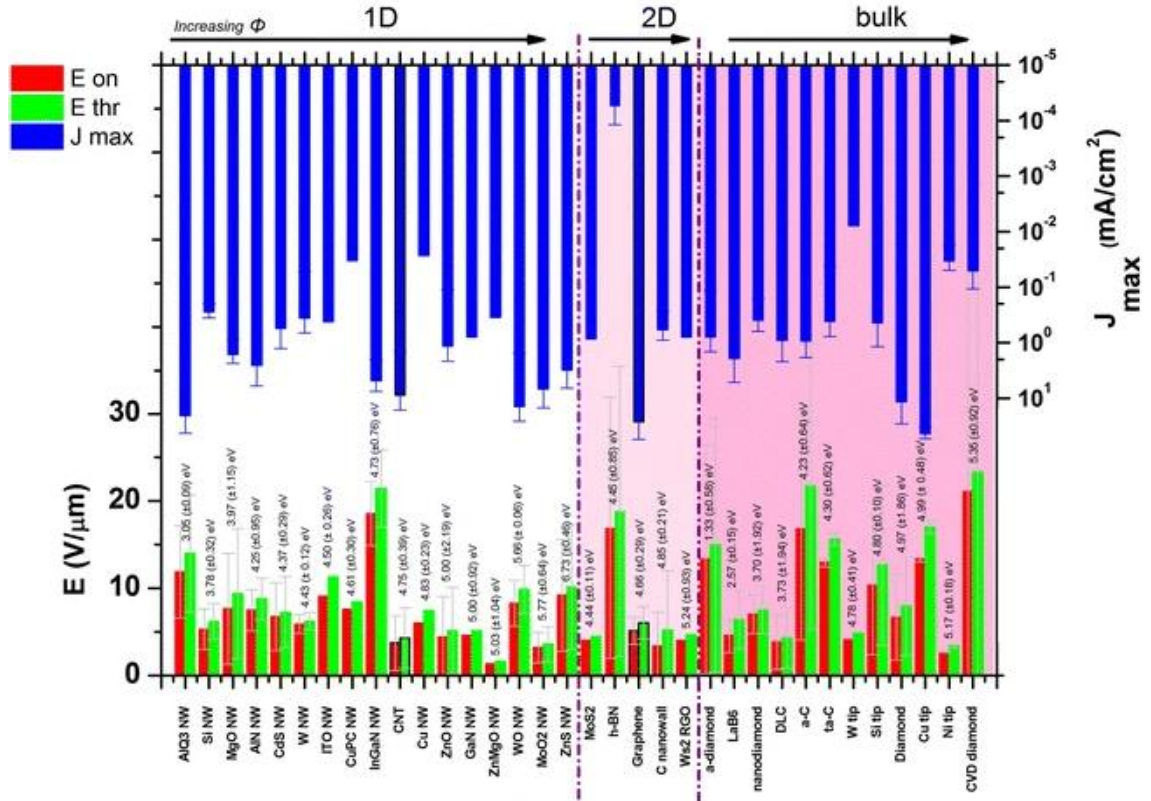


Figure 3.1 Mean turn-on and threshold electric fields and maximum field emission current densities reported in the literature for various FE materials. The x-axis is ordered by increasing dimensionality and work function from left to right. Adapted and reprinted from [13]. Licensed under a Creative Commons Attribution 4.0 (International License: <https://creativecommons.org/licenses/by-nc-nd/4.0/>).

Note that there is no clear correlation between work function, current density, or operating field strength due to the deliberate comparison of emitters with varying morphologies, both within and between materials. As a result, significant differences in the respective FEFs of the emitting structures occur. This also aligns with the findings reported by *Ławrowski* [117]. In his dissertation, he compared the maximum currents emitted by a variety of semi-conducting and low-dimensional materials with different morphologies in diode configuration measurements. He concluded that the conductivity, morphology, and packing density of the emission sites have a greater influence on the maximum achievable emission currents than the material's work function. Note that many of the emitters compared in Figure 3.1 and *Ławrowski's* study can only be realized with a high technological effort and are therefore unsuitable for technical applications, at least for the time being. However, carbon nanotubes (CNTs) in particular have had a prominent presence in the field emission literature for some time, and their fabrication techniques have advanced significantly, making them increasingly established technologies. Due to their exceptional electrical conductivity, thermal stability and aspect ratio, CNTs provide excellent prerequisites for field emitters [113], [115], [118], [119]. Along with the very similar but higher dimensional graphene, they offer one of the

best compromises between high current density and low operating fields of all the materials shown in Figure 3.1. For these reasons, CNT-based field emitters will be discussed in more detail here.

It has been shown that a single multi-walled CNT is capable of carrying 10 mA of dc current, corresponding to a current density of more than 10^9 A/cm², due to their nanometric footprint [120]. In the case of field emission, they can typically be operated at currents up to 10 μ A (sometimes even 100 μ A [121]), but should be kept below 1 μ A for stable emission [122]–[124]. A common failure mechanism of CNT cathodes is the detachment of individual CNTs from the substrate in the presence of strong electric fields. To counteract this, FEAs consisting of islands of densely packed and unoriented CNTs can be used. *Wei et al.* used this method to achieve maximum dc current densities of 4.5 A/cm² (36 mA) measured in diode configuration IV sweeps at a residual gas pressure of $\sim 10^{-6}$ mbar [125]. Current densities in excess of 100 A/cm² have also been reported, but are often due to operation outside of the field emission regime, e.g. by entering the thermally enhanced field emission due to Joule heating of the tip [123], [126]. Nevertheless, this once again proves their durability in terms of current carrying capabilities. *Chen et al.* [127] reported the highest peak emission current of 710 mA (3.55 A/cm²) in pulsed operation by using a similarly patterned array of CNT bundles. The measurement was performed under UHV conditions $8 \cdot 10^{-9}$ mbar using 10 μ s pulses with a 0.1% duty cycle. In summary, it can generally be stated that CNT cathodes in diode configuration typically do not encounter issues with dc emission currents in the few milliamperes range and current densities of several A/cm², as long as reasonable vacuum conditions are maintained [119], [123], [128]–[130].

However, the difficulty with this emitter type becomes apparent when devices are fabricated for triode configuration. Most applications require an integrated extraction electrode, as is the case with Spindt-type cathodes, for example. The electron transmission rate through the gate and current stability is hereby critical, as it impacts both the power consumption of the electron source and the controllability of the anode current. Attempts to integrate extraction gates in CNT cathodes are rare in the literature. This is largely due to the fact, that many of the numerous CNT fabrication techniques are still in development and often lack the reproducibility, compatibility, and precision of common semiconductor manufacturing technologies [13], [119], [131]. Especially the precise alignment of the CNTs in a defined aperture is often challenging [119]. This alignment, however, is crucial to obtaining high electron transmission through the gate. Furthermore, short circuits caused by the bending or detachment of CNTs under lateral electric fields are a common problem in gated devices [119]. Consequently, reported devices in triode configuration typically operate in the mA/cm² range

with small footprints, emitting currents far below milliamperes at comparatively low transmission rates ranging up to 70% with high fluctuations [119], [132]–[137]. Despite these challenges, multiple CNT X-ray sources are currently available for both commercial [138]–[140] and research [9], [22], [141], [142] purposes. Nevertheless, they have not yet achieved significant commercial success, as there is still significant room for improvement of this type of devices [135].

3.2.3 Silicon Field Emitter Arrays

Silicon offers many advantages as a substrate material for field emitters due to extensive research in the semiconductor industry over several decades. The well-established manufacturing processes allow the controlled formation of a wide variety of morphologies on a wafer scale base. These processes also facilitate the incorporation of different insulating and conductive materials to easily create fully functional triode configuration (gated) devices. Furthermore, the conductivity and the conduction mechanism of silicon are comprehensively understood and can be precisely modified through both full substrate and localized doping. This facilitates the integration of various integrated circuits into field emitter arrays using established technology. The following section presents relevant approaches from the literature that may be applicable to this work.

Recently, *Velasquez-Garcia et al.* took advantage of the advances in nanotechnological fabrication techniques to form arrays of well-defined vertical silicon nanowires (SiNWs) that act as current limiters for the emitting tips at the top [143]–[145]. The authors proposed, that high-field carrier-drift-velocity saturation in silicon [146], [147] in combination with the high aspect ratio of the nanowires causes a current-source-like behavior of the emitter resembling ungated field-effect transistors. The current saturation level of these vertical current limiters can be modified by doping. Thus, reducing the substrate’s dopant concentration results in a more pronounced limitation of the emission current and has the potential to extend the lifetime of FEAs. *Huang et al.* took a similar approach by integrating a nanochannel as a serial resistor in their emitter, which was shown to limit the current and heat flow in the tip [148]. While the electric resistance suppresses overloading of individual emitters by limiting the charge carrier supply from the bulk, the heat resistance leads to an increased temperature at the apex, triggering thermally-assisted field emission. However, high temperatures can reduce the ballasting effect of the nanochannel due to decreased impurity scattering probability of the electrons at high thermal velocities [71]. They later improved this technique by implementing p-type silicon as the substrate material [112]. It was hypothesized that the p-type doping of the nanochannel devices restricts the transportation of thermally generated holes

from the tip's apex to the bulk. This, in turn, increases the recombination of electron-hole pairs in the apex, which effectively reduces the net thermal generation of emittable electrons. This negative feedback enhances with increasing temperature, resulting in superior self-modulation at elevated temperatures compared to n-type nanochannel emitters. Other groups have successfully demonstrated emission current control by integrating metal-oxide-semiconductor field-effect transistors (MOSFET) as ballasting elements into the charge carrier supply path of individual field emitters [149]–[152] or entire arrays of emitters [153], [154]. However, this usually results in a limited packing density of the tips, especially if each tip is to be controlled individually. Integrated reverse-biased p/n junctions have been shown to limit the emission current while reducing the noise [155], similar to the effect of using p-type substrates [67], [70], [75], [156]. All these current limiting methods have in common, that they typically lead to a reduction of the maximum possible current density of the array. For this reason, there is always a trade-off here between high emission current densities due to well-conducting substrate materials (e.g. highly doped silicon) and homogeneous current distributions by deliberately limiting the local current densities and potentially extend the FEA's lifetime.

For many years, the benchmark for silicon FEAs was set by *Temple* in 1998 using anisotropically etched tip-on-post emitters with an integrated gate [157]. She reported on a maximum integral dc constant current of over 20 mA for 30 min at 96 V for 28,074 tips in a 0.94 mm^2 array, resulting in a current density of 2.13 A/cm^2 . The measurements were carried out in a vacuum test station with a base pressure of $4 \cdot 10^{-9}$ mbar. Shortly before, she reported on emission currents of almost 10 μA from a single tip fabricated and measured in a similar manner [158]. Even higher integral currents were demonstrated by *Fomani et al.* [159] using a large array of 320,000 titanium and platinum coated silicon tips with an integrated extraction grid on an area of 0.32 cm^2 . The measurement was performed in pulsed mode with a 0.1% duty cycle and 100 μs pulses. The FEA was pretreated by bakeout at 180°C for 24 h. The chamber base pressure was initially about 10^{-9} mbar, but increased to 10^{-7} mbar during the first 5 minutes of operation due to outgassing. An integral current of 350 mA was achieved at an extraction voltage of 300 V. This gives an average maximum current of 1.1 μA per tip and a maximum current density of 1.1 A/cm^2 . The highest current density has recently been demonstrated by *Guerrera et al.* using low dimensional silicon nanowire emitters fabricated using lowly n-doped silicon substrates [160]–[162]. Based on the work of *Velasquez-Garcia et al.* [143]–[145], the nanowires are intended to act as current limiters in order to ensure that the current load is distributed as uniformly as possible across the array. They are embedded in a dielectric matrix and are provided with self-aligned gate apertures using a

highly sophisticated fabrication process. All the measurements were performed in dc mode triode configuration at a base pressure of $\sim 3 \cdot 10^{-10}$ mbar. During IV sweep measurements, *Guerrera* was able to show, that the FEAs typically emit up to 1 μA per tip, more or less independent of the size of the array [160]. The maximum current density reached above 100 A/cm^2 during the sweeps, typically at extraction voltages between 50 V and 60 V. This value was calculated using the 1 μm^2 footprint of a single tip including its surrounding gate. Notably, they also showed significant lifetime and stability. A single emitter at 1 μA (100 A/cm^2) could be operated for over 100 h without noticeable degradation under UHV conditions at $3 \cdot 10^{-10}$ mbar. Arrays of 100 x 100 and 1000 x 1000 emitters, operated at a constant current emission of 1 mA (equivalent to 10 A/cm^2 and 0.1 A/cm^2 , respectively), also survived over 100 h, however, with a slight increase in voltage to maintain the current. Based on these findings, initial research has been conducted to incorporate the gated FEAs into actively pumped X-ray sources for X-ray computed tomography and planar X-ray imaging [163].

3.3 Conclusion

The purpose of this chapter is to discuss the challenges associated with interpreting field emission data. Even under ideal experimental conditions, connecting theory with experiment can be challenging. The accuracy of the data is inevitably influenced by various uncertainties and theoretical assumptions, which may not hold in practice. Therefore, it is advisable to exercise caution when interpreting the absolute values of extracted data, such as the FEF and effective emission area, especially when dealing with multitip emission from FEAs. Apart from that, the circumstances under which the data were collected are just as significant as the theory used for evaluation. The performance of FEAs is significantly affected by the experimental conditions, such as operating pressure, residual gas composition in the measurement chamber, operating mode, or pretreatment of the emitter. Therefore, even directly interpretable data, such as the maximum emission current and the maximum current density, can only be compared if they are acquired under the same experimental conditions. Unfortunately, the literature often lacks detailed information on experimental conditions, making it challenging to make objective comparisons of results among different research groups.

Despite these challenges, a thorough review of the current state of the art shows that many FEAs made from a variety of materials show promise in meeting the requirements of high-current applications, at least under UHV conditions [123], [164]. The Spindt-type emitters were found to exhibit the highest reported current density of 1000 A/cm^2 [85] in UHV and pulsed operation. However, there is a lack of empirical data regarding their lifetime under

constant current operation at elevated ambient pressures. CNTs demonstrate promising potential as FE-based electron sources, as a result of their exceptional electrical and thermal durability, as well as their inherently high aspect ratio morphology. In diode configuration IV sweeps, this enables them to provide emission current densities up to the A/cm^2 range even under non-UHV conditions [125]. Unfortunately, integrating a high-transmissivity gate electrode with sufficient device lifetime remains challenging due to limited process reproducibility, compatibility, and precision compared to well-established semiconductor technologies. Recent research suggests that sophisticatedly fabricated silicon FEAs have the potential to produce similar results. It has been reported that current densities of $100 \text{ A}/\text{cm}^2$ (for $1 \text{ }\mu\text{A}$ emission current) and emission currents of 1 mA (with a current density of $10 \text{ A}/\text{cm}^2$) can be attained for more than 100 hours in dc operation under UHV conditions [160]. This, together with the simple gate integration via standard semiconductor technology, demonstrates significant potential in implementing these devices in a portable X-ray tube, as envisioned here. The remaining challenge is to ensure the lifetime and emission stability of the emitters under adverse conditions far beyond UHV. For this reason, the following chapters deal with the development and characterization of new silicon FEAs based on the knowledge from the literature. Notably differing from literature, the FEAs are evaluated under realistic conditions, specifically at a pressure of 10^{-5} mbar and in constant current operation, which corresponds to our actual application requirements.

4 Methods

This chapter presents the methods that are necessary for the development and characterization of the various FEAs, which will be discussed comprehensively in the subsequent chapters. All the devices were fabricated at the laboratories of the University of the Bundeswehr Munich, while electrical characterization was performed in custom-built measurement setups at KETEK GmbH Munich.

4.1 Key Technologies for Device Fabrication

Two types of FEAs are presented in this thesis, which differ fundamentally in their morphology. Both manufacturing processes are based on different key technologies, which will be presented in more detail below.

4.1.1 Orientation Dependent Wet Chemical Etching of Silicon

The first key technology is the orientation dependent or anisotropic wet chemical etching of silicon. When silicon reacts with strong alkaline solutions, it exhibits variable etch rates that depend on the crystallographic orientation of the exposed surfaces. The selectivity between different crystal planes is thereby a function of the composition, temperature and concentration of the etchant [165]. The most commonly used etchants in the semiconductor industry are potassium hydroxide and tetramethylammonium hydroxide (TMAH). The latter has the advantage of CMOS compatibility and is therefore used in this work for anisotropic silicon etching [166]–[168].

The wet chemical etching behavior of silicon is determined by the orientation of unmasked surfaces in relation to the crystal lattice and the geometry of the structure to be etched. Figure 4.1 illustrates how the orientation of rectangular openings in the masking layer affects the etching profile during wet chemical silicon etching. Alignment of the aperture along the $\langle 110 \rangle$ direction typically results in V-shaped trench profiles for $\langle 100 \rangle$ silicon wafers. This profile is dominated by the particularly slow etching $\{111\}$ planes, which act as an etch stop for the erosion of the $\{100\}$ surface [166], [169]–[171]. A characteristic of this process is the included angle of exactly 54.7° between the $\{100\}$ and the $\{111\}$ plane, as well as the almost atomically smooth surface of the $\{111\}$ planes [166]. Rotating the opening by 45° , however, results in the $\{111\}$ planes being tangential to the concave corners, making them unable to form a stable etch stop plane. As a result, the shape of the trench is dominated by either the

also slowly etching $\{100\}$ or $\{110\}$ planes, depending on the etchant and etching parameters [165], [169], [170].

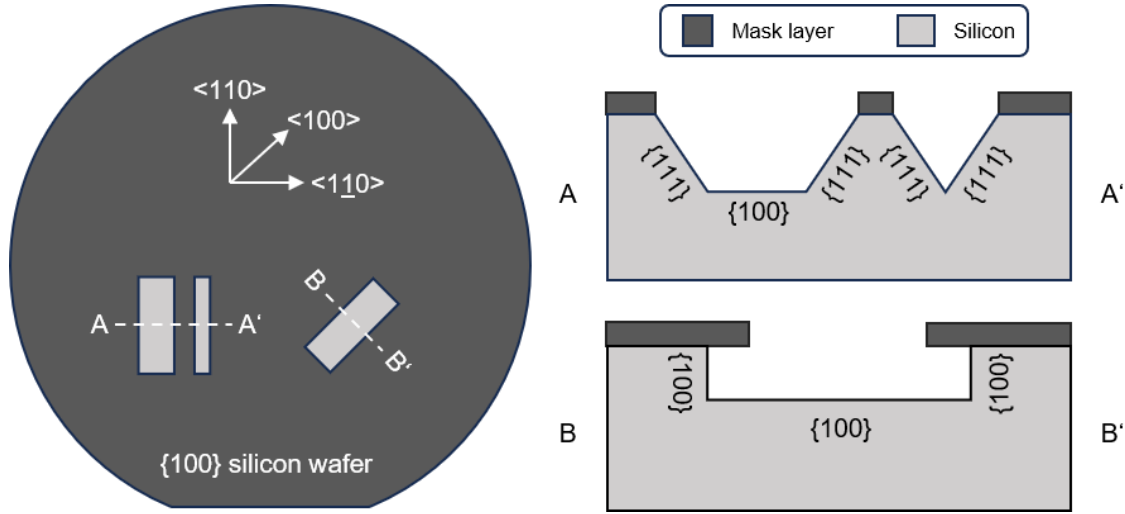


Figure 4.1 Schematic etch profiles for differently oriented mask apertures on a $\langle 100 \rangle$ silicon wafer after wet chemical etching. Adapted from [172].

The etching behavior of mask openings with concave corners is therefore primarily determined by the slowest etching exposed surfaces. In contrast, when etching convex corners, the fast-etching planes dominate the etching profile, as under-etching of the exposed surfaces prevents the formation of stable etch stop planes [165]. A very detailed overview of the characteristics and differences in etching concave and convex corners can be found in the work of *Pal and Sato* [165]. The crucial aspect of convex corner etching for this work is that the etching parameters determine which etching planes will ultimately prevail. This will be utilized in Chapter 5.1 to create pyramid-shaped silicon tips based on a maskless, anisotropic etching process.

4.1.2 Reactive Ion Etching

The second key technology in this work is reactive ion etching (RIE). RIE belongs to the plasma-based dry etching techniques and its working principle is quite similar to sputter etching. The most basic RIE systems typically consist of a vacuum chamber containing two parallel plate electrodes of different sizes and a gas supply system. The larger upper electrode is grounded, and the smaller lower electrode is capacitively connected to a high-frequency (HF) power source. A plasma is ignited between the plates by coupling HF power into the residual gas at low pressure. This plasma contains free electrons and ions that oscillate in the alternating field. The ion current is significantly lower than the electron current due to the substantial mass difference between these two types of charged particles [173], [174]. This leads to self-biasing, which sustains the plasma. In more advanced systems, a second power

source can be coupled inductively into the plasma via an HF coil. Figure 4.2 illustrates a schematic cross-section of such an inductively coupled plasma reactive ion etching system (ICP RIE), which is also used in this work.

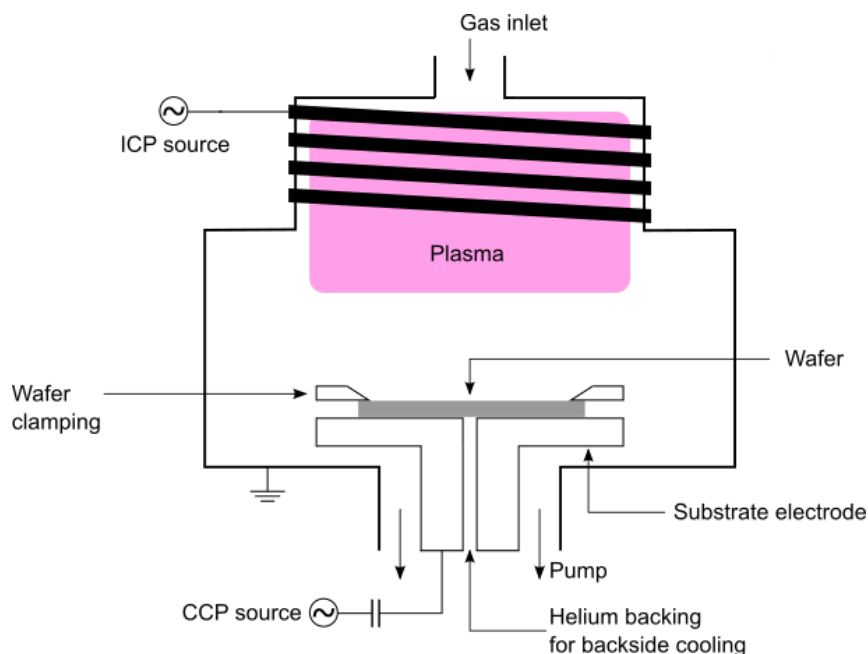


Figure 4.2 Schematic cross-section of an inductively coupled plasma reactive ion etcher, including the inductively coupled plasma (ICP) source and the capacitively coupled plasma (CCP) source.

The vacuum chamber, which is grounded, serves as the upper electrode in this case, opposing the capacitively coupled substrate electrode. The HF coil is typically located at the top of the chamber. Its purpose is to induce a magnetic field into the plasma, which accelerates the charged particles onto a circular path. This increases the residence time and distance traveled by the particles in the plasma, which enhances the probability of interaction with other particles and thus the plasma density [174]. The wafer is clamped onto the substrate electrode in the reaction chamber. To facilitate cooling or heating during the process, a helium backing is used for the thermal contact between wafer and electrode in the evacuated chamber. The intended gas or gas mixture is introduced into the chamber via the gas inlet.

Regardless of the type of RIE system, material removal during etching is always based on two different processes. Firstly, ions from the plasma can be accelerated onto the substrate, primarily due to the area ratio of the electrodes [173]. The kinetic energy of the ions is typically in a range between 0.1 and 1 keV, which exceeds the usual binding energies of solids by several orders of magnitude [173]. As a result, physical ablation occurs when the ions collide with the substrate. This ablation is mainly anisotropic due to the directional nature of the accelerated ions. Secondly, unlike sputter etching, RIE etching uses a reactive gas as the basis of the plasma. This leads to the generation of reactive ions and radicals in the plasma,

which can cause chemical erosion of the substrate material. Therefore, RIE etching involves both physical anisotropic and chemical isotropic etch mechanisms. Which part dominates the etch profile is primarily determined by the plasma density and the ion energy, which are both affected by various multilaterally intertwined process parameters. However, the advantage of ICP RIE systems as opposed to conventional ones is the availability of two distinct power sources to excite the plasma. Simply put, the ICP source primarily regulates the plasma density, which affects the quantity of reactive particles present in the reaction chamber and thus the chemical erosion [171], [174]. In contrast, the capacitively coupled power strongly influences the energy of the ion impact. The interaction between the two powers, along with the gas flows, substrate temperature and chamber pressure, allows for adjustment of the ratio between chemical erosion and physical ablation during RIE etching, which in turn affects the etch profile [171], [174].

In this work, the ICP RIE system is exclusively used for deep reactive ion etching (DRIE). DRIE describes a group of plasma-based dry etching techniques, which are used for highly anisotropic etching in microsystems technology. One of the most prominent representatives for DRIE of silicon is the Bosch process, which was first developed and patented by the Robert Bosch GmbH in the 1990s [175]. It consists of an alternating sequence of isotropic passivation and semi-anisotropic etching of silicon due to a combination of physical ablation and chemical erosion. The sequence of this process is schematically illustrated in Figure 4.3.

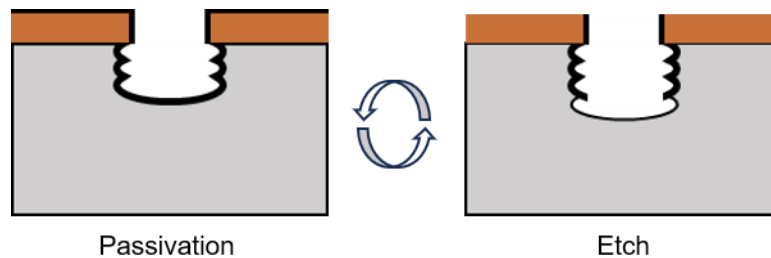


Figure 4.3 Schematic illustration of the etch and passivation step during the Bosch process.

In the herein used process (see Appendix A, Table 3, Table 4 and Table 5), the sequence begins with an initial ignition of the plasma, followed by the passivation step. By introducing C_4F_8 into the plasma for a duration of 5 seconds, a fluorocarbon layer is isotropically deposited on the substrate's surface, including both the photo resist and the exposed silicon. The passivation layer is chemically inert and shields all surfaces from reactive particle erosion [176], [177]. During the following etching step, SF_6 is introduced into the reaction chamber to obtain the reactive fluorine particles required for chemical etching of silicon. Typically, the conductively coupled power is increased for this step to enhance the physical ablation. As mentioned before, the physical ablation by the ions is highly directional, resulting in removal

of the passivation from horizontal planes only, while vertical surfaces remain passivated. Once the surface is exposed, the plasma's fluorine radicals start to isotropically etch the silicon in addition to the still ongoing anisotropic physical ablation. The cycle then starts anew, etching deeper into the wafer with each iteration, allowing for a vertical etch profile through suitable parameter selection and fast cycling. A certain roughness of the sidewall is unavoidable, which becomes noticeable through ripples. The depth of these ripples can be controlled by the duration of the etching step in the alternating process.

4.2 Electrical Characterization Methods

Two custom-built measurement setups are used to characterize and measure the silicon FEAs fabricated in this dissertation. The first setup is utilized to perform electrical characterization in both diode and triode configurations, while the second was specifically constructed to measure the emission current distribution of FEAs using a newly developed evaluation method based on a Raspberry Pi HQ camera [178]. The basic electrical circuitry and measurement chamber design is similar in both cases. Therefore, this section concentrates solely on the measurement setup for the pure electrical characterization to prevent redundancy. A more detailed description of the special features of the second setup can be found in Chapter 7.2.3. Apart from the introduction of the measurement setup, this chapter illustrates and discusses the different measurement procedures used for electrical characterization in this work along with the basic evaluation principles.

4.2.1 Measurement Setup and Specimen Holder

Figure 4.4(a) shows a photograph of the measurement setup used for electrical characterization, accompanied by a photograph of the specimen holder in Figure 4.4(b) and a schematic diagram of the specimen holder in side view in Figure 4.4(c). The core part of the measurement setup shown in Figure 4.4(a) is a KF-40 cross, which acts as the measurement chamber (A). To create the necessary vacuum for measuring the field emitters in the chamber, a turbomolecular pump with an integrated backing pump (B) is attached to the lower flange of the recipient. A T-piece containing a pressure gauge (C) and an electromechanical needle valve (D) is mounted on the upper flange. Nitrogen with 99% purity can be injected into the recipient via the valve. Together with the pressure gauge and a digital proportional-integral controller, constant pressure levels between $1 \cdot 10^{-3}$ and $5 \cdot 10^{-7}$ mbar can be set in the chamber. A vacuum feedthrough (E) allows the specimen holder to be inserted into the recipient from the right side. This is connected to the regulation circuit (F) and one of the two high voltage power supplies (G), which has a maximum extraction voltage of 2 kV.

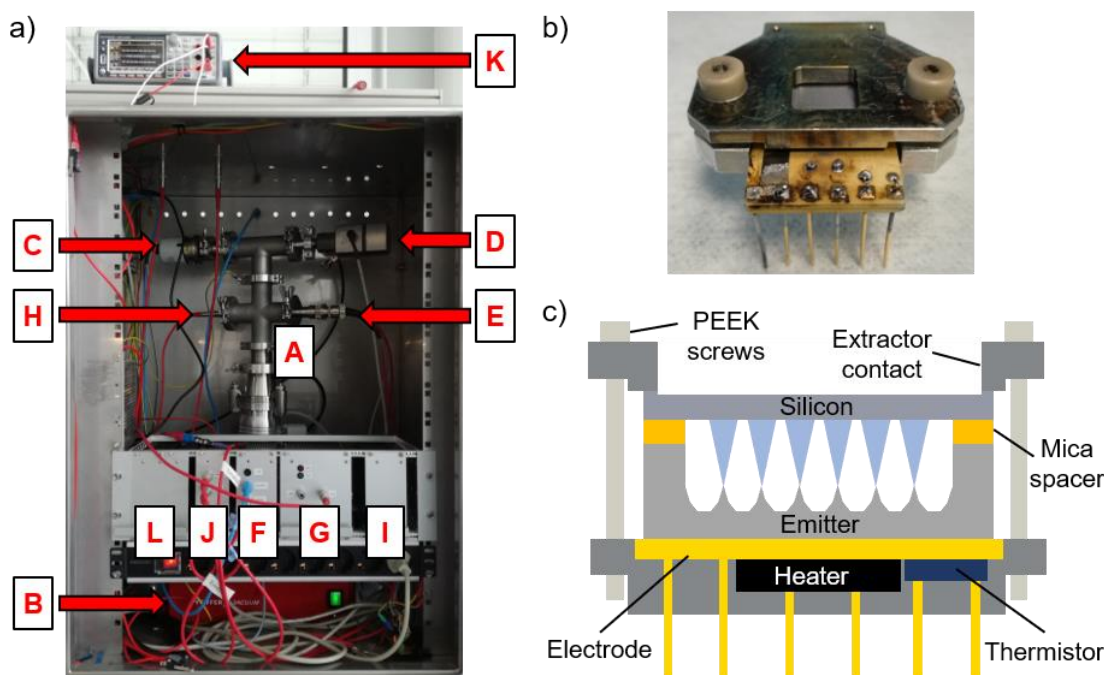


Figure 4.4 (a) Photograph of the Measurement setup, showing the measurement chamber (A), the turbomolecular and backing pump (B), the pressure gauge (C), the electro mechanical needle valve (D), the vacuum feedthrough of the sample holder (E), the regulation circuit (F), two high voltage power supplies (G), the vacuum feedthrough for the anode (H), the control board with microcontroller (I), the high impedance resistor (J), the digital multimeter (K) and the protection circuits (L). (b,c) depict a photograph and a schematic of the specimen holder used for measurements in diode configuration. Adapted from [179].

An anode (H) can be inserted from the left side for transmission measurements in triode configuration. The anode voltage is limited to a maximum of 6 kV by the second high voltage power supply (G). A 16-bit microcontroller (Amtel ATx-mega256A3BU [180]) regulates the measurement setup. It is mounted on control board (I) alongside an external digital-to-analog converter (DAC) [181] and an analog-to-digital converter (ADC) [182]. These external devices offer a greater resolution compared to the internal DACs and ADCs of the microcontroller. The measuring PC, which is not visible in Figure 4.4(a), connects with the microcontroller through an I²C connection. In current-controlled measurements, the voltage drop across the control circuitry dictates the extraction voltage applied to the emitter. To measure this accurately, a high-impedance resistor (J) was incorporated into the circuit (see Chapter 4.2.2). The emission current is measured with a multimeter GDM9061 (K) [183], which is capable of measuring with minimal voltage interference. For safety, all signal paths from the lockable measurement setup pass through protection circuit (L), which is equipped with gas discharge tubes, varistors, and suppressor diodes. These components filter out any possible high-frequency arcs to the ground through their parasitic capacitances. They also prevent the

measurement PC from communicating with the microcontroller via USB, so this is done instead via a Universal Asynchronous Receiver Transmitter circuit, which is translated to USB after the high voltage protection and transmitted to the PC.

The sample holder shown in Figure 4.4(b,c) comprises an aluminum frame and a ceramic plate. On the front of the ceramic board, there is a gold electrode, while a heating component and thermistor for temperature measurement and control are located on the rear. These components interface with gold pins via the vacuum feedthrough. For diode configuration measurements, the FEA is placed on the gold electrode, along with a mica spacer, which is intended to provide electrical insulation between the extractor and the emitter. The thickness of these typically ranges between 25 μm and 60 μm with a nominal dielectric strength of 0.118 kV/ μm [184]. Deviating from the theoretic breakdown voltage defined by this, parasitic creepage currents across the insulator surface can already occur at lower voltages during actual measurements. The extent of these currents depends on the measurement conditions, as the surface states are affected by the ambient pressure and the residual gas composition. Empirical investigations suggest that leakage currents are adequately suppressed under typical measurement conditions for voltages up to 1.5 kV at mica thicknesses of approximately 30 μm and higher. Consequently, an estimate of roughly 0.05 kV/ μm is more realistic considering leakage currents. A silicon plate is subsequently added on top of the mica, serving as the planar extraction anode for a homogeneous macroscopic field distribution over the array. In triode configuration measurements, the mica spacer and silicon plate are omitted because the extractor is already integrated on the emitter (see Chapter 6). Here, the external anode (H) acts as the collector of the transmitted current through the integrated extraction gate. In both configurations, the assembly is enclosed between two precision machined aluminum frames, which are joined by two polyetheretherketone (PEEK) screws for proper insulation. The upper component features a cutout to allow for electron transmission during triode configuration measurements. It is electrically connected to the vacuum feedthrough using gold pins that pierce the ceramic plate. In diode configuration this component is in direct contact with the silicon plate. Therefore, the gold pins can be used to set the extractor potential via the vacuum feedthrough. For triode measurements, it is necessary to ensure that the integrated extraction gate can also be addressed through physical contact with the upper part of the aluminum frame.

4.2.2 Circuitry

Figure 4.5 schematically depicts the complete circuitry of the measurement setup. As previously stated, the setup includes a current regulation circuit, indicated in yellow in the

figure. This is based on a publication by *Prommesberger et al.* [185] and has been shown to be able to control the emission current with fluctuations of less than 1%, ensuring a stable output.

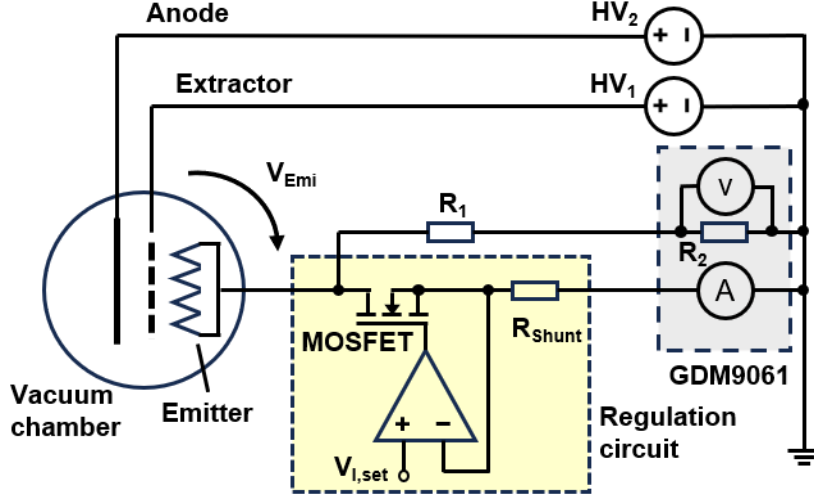


Figure 4.5 Schematic representation of the measurement circuit, with the current regulation circuit highlighted in yellow.

For each measurement, a specific potential is applied to the extractor using the high voltage source HV_1 to stimulate the FEA to emit. The present measurement setup limits this potential via the installed MOSFET, which is designed for drain-source voltages of up to 1.5 kV. To avoid a component defect, the maximum extraction potential applicable in this work is therefore limited to 1.4 kV. When taking transmission measurements, an additional potential of typically a few kV is applied to the anode by the second high voltage source HV_2 . In diode configuration, this is simply set to zero, as the extractor acts as the anode in this case.

The control circuit, situated along the emitter's current path, has two potential functions. Firstly, it can limit the maximum current emitted by the FEA to prevent sudden current peaks that could pose risks to the measurement setup and emitter. Secondly, it allows the FEA to operate at defined current levels, if the extractor potential is sufficient. Its operating principle is based on a MOSFET whose gate is controlled by an operational amplifier (OPAMP) in a comparator configuration. The OPAMP operates within a negative feedback loop that connects to the shunt resistor R_{shunt} , while the MOSFET manages current flow into the FEA. By setting a specific voltage, $V_{I,set}$, at the non-inverting input of the OPAMP the maximum current through the shunt resistor R_{shunt} can be limited. This maximum current corresponds to the maximum emission current from the emitter and can be calculated according to the relationship $I_{Emi,max} = V_{I,set}/R_{shunt}$. If the extractor potential remains low enough to prevent the emission current from exceeding its current level, the OPAMP produces a positive output and the MOSFET remains fully open. Once the control current is reached, the OPAMP closes the

gate of the MOSFET to maintain the set current limit. Any excess voltage of the extractor potential drops across the MOSFET. The voltage range for $V_{I,\text{set}}$ is constrained from 10 mV to 15 V due to the DAC's amplifier circuitry. To accommodate emission currents ranging from a few nanoamperes to several milliamperes, a relay is used to choose among three shunt resistors with values of 3.3 M Ω , 59 k Ω , and 1.05 k Ω .

To reconstruct the emission voltage V_{Emi} that is responsible for the emission current, it is necessary to measure the voltage drop across the control circuit. This is done using a high resistance voltage divider consisting of $R_1 = 500 \text{ G}\Omega$ and the internal resistance $R_2 = 10 \text{ M}\Omega$ of the multimeter. This was calibrated once during installation. The maximum error current through the voltage divider can be estimated to 2.8 nA, based on *Ohm's law*, the combined series resistance $R_1 + R_2 = 500.01 \text{ G}\Omega$ and the maximum extractor voltage of 1.4 kV. This value is multiple magnitudes lower than the targeted emission currents. Nevertheless, in all evaluations, the measured emission current is adjusted by subtracting the fault current associated with the corresponding voltage drop across the control circuit.

4.2.3 Current-Voltage Measurements

The most common technique in the literature for evaluating field emitters are IV sweeps. In the basic version, the extraction voltage is gradually increased, and the emitted current from the FEA is measured accordingly. However, this approach suffers from significant comparability issues when applied to different types of emitters. For instance, when measuring in diode configuration, the distance from the tip to the anode can vary significantly among various FEAs and setups. This, in turn, affects the electric field and renders comparing operating voltages and maximum currents meaningless. Even worse is the significant divergence in current load among emitters at fixed voltage steps attributable to their varying intrinsic operational ranges. This is particularly relevant for lengthy measurements under harsh environmental conditions. To avoid this issue, only current-controlled IV sweeps are used in this work. For comparative measurements of different FEA types in diode configuration, the macroscopic electric field applied to the emitter is always included. The calculation of the electric field is based on the ratio of the vertical distance between the extraction electrode and the apex of the emitting tip and the extraction voltage. For all IV characteristic measurements, typically 21 data points are collected per sweep, with the set currents distributed linearly along a logarithmic scale. To account for the maximum error current of 2.8 nA mentioned earlier, the minimum current is fixed at 3 nA. The upper current limit can be chosen at will. Four measurements are taken at each set current level (each data point) for every individual

sweep. The emitted current, applied extractor and anode voltage, voltage drop across the internal resistance R_2 of the voltage divider, present vacuum chamber pressure, and sample holder temperature are recorded.

Figure 4.6(a) displays the results of a typical IV characteristics measurement in diode configuration, plotted in standard semi-logarithmic form. The measurement was performed with a maximum current of 10 μA , and six individual sweeps (three up, three down) were conducted. The orientation of the triangular marker indicates the direction of each sweep. Each data point corresponds to the average value of four measurements and the error bars are derived from the empirical standard deviation. As mentioned in Chapter 2.4, field emission measurements are prone to misinterpretation, if the origin of the measured current is not verified. Therefore, it is always advisable to plot field emission IV characteristics in a linearized form. The most accurate mathematical and physical approach is to use Murphy-Good coordinates [31], [33], as shown in Figure 4.6(b). According to Equation (2.11) this plot results in a perfectly linear course of the IV characteristics if the measured current originates of an ideal FE system. However, neither the emission current nor the corresponding extraction voltage can be read directly from this type of plot. In contrast, data representation in Millikan-Lauritsen coordinates [78], as shown in Figure 4.6 (c), is much easier to interpret, as here the emission current is plotted directly in logarithmic form on the y-axis. A comparison of the two plots reveals that the theoretically not perfectly linear characteristic in ML coordinates can hardly be distinguished from that in MG coordinates for real measurement data [186]. Nevertheless, direct reading of the measurement data from the plot is still hindered by the inverse voltage axis. To address this issue, the x-axis can be rescaled reciprocally, relabeled with the actual voltages instead of its inverse, and mirrored. As shown in Figure 4.6(d), this enables direct reading of the emission current and corresponding extraction voltage, while also allowing for data verification via the linearity of the characteristics in ML coordinates. Throughout this thesis, the adapted Millikan-Lauritsen plot will be the exclusive method to present IV measurement data.

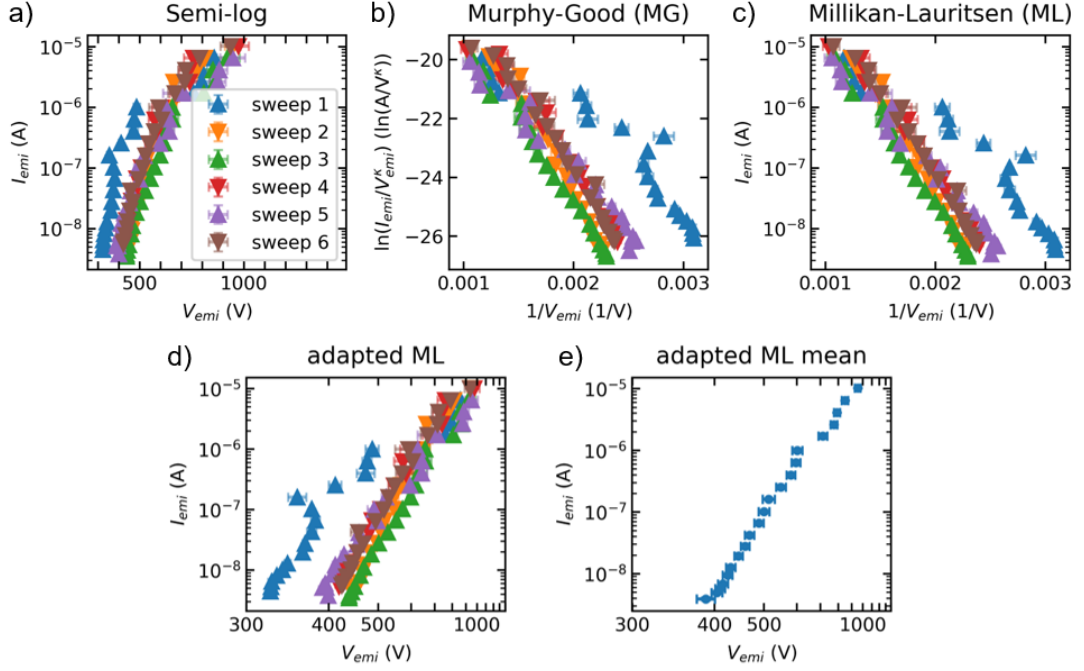


Figure 4.6 Typical current-controlled IV characteristics presented in different coordinate systems. The individual sweeps are displayed in (a) standard semi-logarithmic coordinates, (b) Murphy-Good coordinates [31], [33], with $\kappa = 1.1853$ (see Chapter 2.1), (c) Millikan-Lauritsen coordinates [78], and (d) adapted Millikan-Lauritsen (ML) coordinates. The orientation of the triangular markers indicates the direction of the respective partial sweep. For the adapted ML coordinates, the x-axis is reversed, reciprocally rescaled, and relabeled with non-reciprocal voltages to increase readability of the plot. (e) depicts the corresponding mean characteristics in adapted ML coordinates.

In addition to the various forms of representing IV characteristics, this example measurement can be used to discuss a field emission phenomenon, which will frequently recur in this work. The initial sweep displayed in blue in Figure 4.6(a-d) demonstrates typical conditioning effects of the initially operated FEAs, which have been comprehensively documented in the literature [83], [187], [188]. The first measurement points or initial few sweeps tend to vary significantly from the rest of the data points. This divergence can be in the direction of both higher and lower extraction voltages and can be explained by multiple factors. One possible cause is the presence of exceptionally sharp tips in the array, which can cause strong emissions at low voltages during the first sweep of the measurement. If this current is carried exclusively by a few tips, the resulting high local current densities can lead to their degradation. To maintain the set emission current, the voltage must be raised during subsequent sweeps to account for the lack of sharp emitters and allow for compensation by duller tips [143]. Another factor to consider is the impact of adsorbates on the surface of the emitter. The emission of electrons causes Joule and/or Nottingham heating of the emitter, leading to an increase in adsorbate desorption [55], [56]. After a certain amount of time under consistent

conditions, the temperature will eventually stabilize, resulting in an equal balance of adsorption and desorption [83]. This allows then for the establishment of a stable characteristic curve. Also, the native oxide on the surface of silicon may have a potential impact. For instance, the emission of hot electrons can result in the formation of conductive channels within the oxide, thereby increasing the emission current at a constant extraction voltage [189]. Over time, the oxide, including the conductive channels, can be removed from the emission region, e.g., by ion bombardment. This would explain the enhanced emission at lower voltages only for the first sweep. Alternatively, the natural oxide can also initially inhibit the emission, which causes a shift in the characteristic curve to higher voltages. As the oxide is removed, the characteristic curve then shifts to lower voltages until it reaches a stable level.

In literature, it is common practice to exclude sweeps that display noticeable behavior of this kind during data processing [3], [190]. However, the number of sweeps to exclude is subjective and arbitrary. Figure 4.6(e) displays the averaged characteristics for the entire measurement. Here, all corresponding data points from all the sweeps were averaged and assigned a measurement uncertainty based on the empirical standard deviation for each set current level. The large number of individual sweeps typically allows for disregarding deviations in the initial sweep during averaging. Therefore, for this work, every sweep is included in calculating the mean characteristics.

4.2.4 Current Dependent Lifetime Test

In addition to the emitter's characteristics, its lifetime under specific environmental conditions is often of interest. The literature frequently estimates this by taking measurements at a constant voltage for arbitrary time periods. However, the electric fields applied to the emitter depends on the specific measurement setup and FEA. Furthermore, the current load experienced by an emitter is random and not comparable across various FEAs. Actual applications typically demand specific emission currents over defined time periods. Measuring at a constant current thus provides more accurate estimations of lifetime. Still, the current level and duration can again be arbitrarily selected, which makes comparing measurements difficult. For these reasons, *Edler* dedicated a significant portion of his dissertation to creating a current-dependent lifetime test (CDLT) for FEAs [191]. This measurement method was initially presented at the *34th International Vacuum Nanoelectronics Conference (IVNC)* in 2021 by our group [192], [193]. Note that while this methodology shows promise in offering novel insights into the current-dependent performance of FEAs, it still requires further validation in many areas before in-depth conclusions can be drawn with sufficient certainty. In this work, it is used to evaluate the potential of the different types of FEAs fabricated to meet

the requirements for application in a portable X-ray tube and give a basis for a comparison of different emitter types. Therefore, the presented studies will primarily focus on the key aspects of the data to avoid unfounded speculations and follow-up measurements beyond the scope of this work. For a more detailed analysis of the method itself and the interpretation of the measurement results, please refer to [191]. This chapter presents a concise summary of the methodology and attainable results, emphasizing the significant aspects for comparing different FEAs.

The test is designed to evaluate the degradation of FEAs at several different current levels. For each level, a three-step sub-measurement is performed. The first step records a current-controlled IV characteristic curve, as outlined in the previous chapter. The maximum current of the sweeps corresponds to the present set current level of the CDLT. Following this, a one-hour constant current measurement (CCM) is conducted at the set current level (maximum current from the sweeps) to examine the lifetime or degradation of the FEA for that specific current level. The measurement is programmed to terminate when the emitter fails to maintain the current control within a range of $\pm 10\%$ for 20 consecutive measurement points. Afterwards, the initial current-controlled IV characteristic measurement is repeated to conclude the measurement, regardless of whether the emitter has sustained the full hour of the CCM or not. If the emitter successfully emitted the set current within the maximum available extraction voltage for the entire hour, the subsequent three-step sub-measurement (IV sweeps, CCM, IV sweeps) will be conducted at the next higher set current level. Otherwise, the entire measurement terminates. The one-hour emission time during CCM was determined as a beneficial trade-off to restrict the overall measurement duration, while being long enough to reach a state of steady and notable deterioration. In prior research, it could be shown that most of the current dependent degradation happens during the first one to five hours of CCM [191], [194].

Figure 4.7 illustrates exemplary measurement results for a single CDLT sub-measurement at a set current of $10\ \mu\text{A}$. The measurement was carried out on a TMAH FEA, which will be presented in detail in Chapter 5.1. In Figure 4.7(a), the emitter's characteristics before and after the one-hour CCM are shown in adapted ML coordinates. The characteristics after the CCM exhibit a significant shift towards higher voltages compared to the initial characteristics. This shift is due to the FEA's degradation caused by the constant current load during CCM, which is also visible in Figure 4.7(b). It depicts the extraction voltage applied to the FEA by the control circuit during the CCM in order to maintain the set emission current of $10\ \mu\text{A}$. In accordance with the voltage shift in the characteristic, the extraction voltage increases continuously with increasing operation time during CCM.

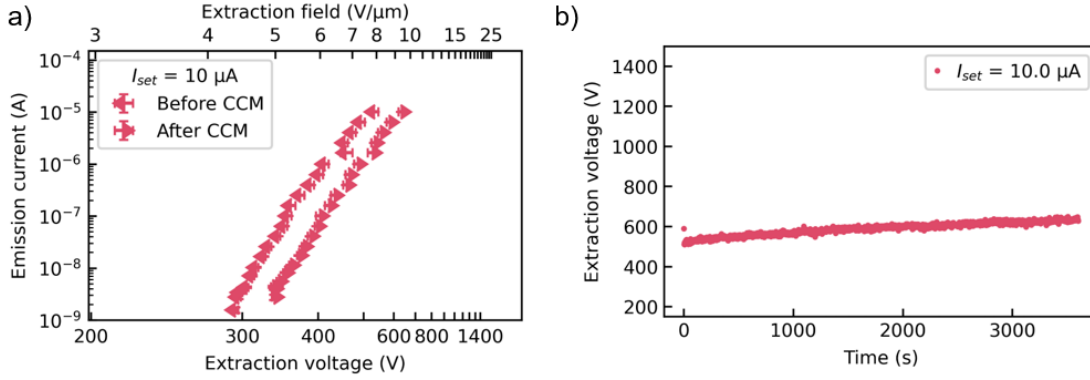


Figure 4.7 Measurement results of a CDLT sub-measurement at a set current level of 10 μA . (a) displays the emitter's characteristics before and after the CCM in adapted ML coordinates. (b) illustrates the temporal evolution of extraction voltage required to sustain the 10 μA during the constant current measurement.

To assess the current dependence of the degradation, Figure 4.8 shows the measurement results for the entire CDLT including all the sub-measurements. The current levels examined in the test are highlighted as white dashes in the color bars of the plots. They are linearly distributed on a logarithmic current axis, similar to the set currents in the current-controlled sweeps. To save measurement time, a resolution of five current levels per order of magnitude was chosen only for currents of 1 μA and above, while below 1 μA only three additional levels are investigated, since significantly less degradation is expected at lower currents. The minimum level of 10 nA was selected due to the restricted current resolution of the measurement setup. Figure 4.8(a) displays the averaged characteristics of the sweeps before and after each CCM in adapted ML coordinates. The dotted lines depict a linear regression, that was carried out in Murphy-Good coordinates for each characteristic. The slope and y-axis intercept of these fits can later be used to determine the respective field enhancement factor and effective emission area as described in Chapter 2.4. Figure 4.8(b) represents the extraction voltage needed to maintain the constant emission current over the temporal course of the CCM for each set current level. The data points for each current level are fitted using linear regression, which is plotted as a solid line in the respective color. This linear regression is only carried out if the CCM lasted for at least 100 data points to prevent unreasonable fits. It will later be used to evaluate the mean degradation over time for each current level.

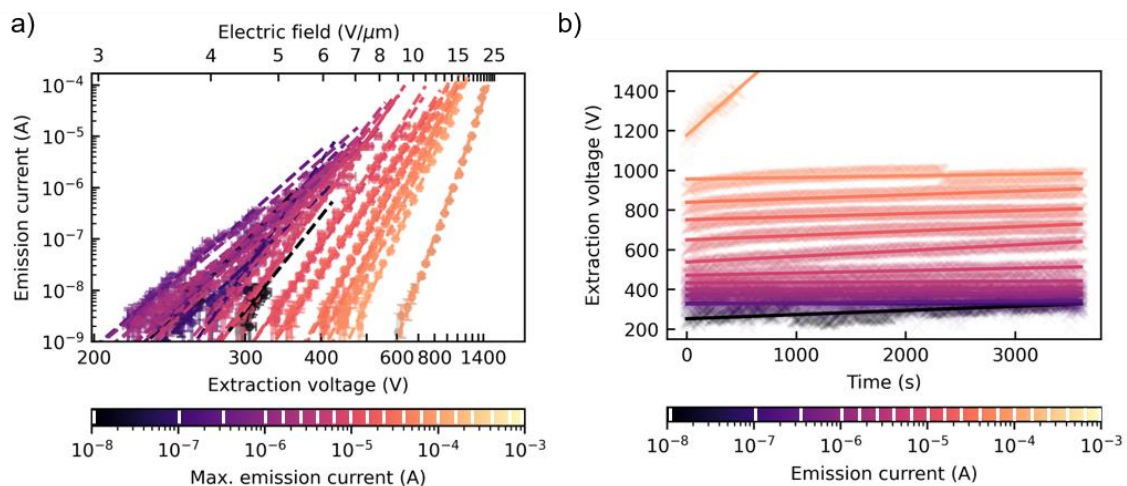


Figure 4.8 Measurement results of the current dependent lifetime test of a single TMAH FEA sample. Both plots use colors to represent the sub-measurement's current level. The distribution of the set current levels is indicated as white dashes in the color bars. (a) displays the characteristics before and after each constant current measurement at every current level in adapted ML coordinates with reciprocal scaled and reversed x-axis. (b) illustrates the temporal evolution of extraction voltage required to sustain the respective set current during the constant current measurement.

By closely examining Figure 4.8(a), two curves can be observed for each color (set current level), representing the characteristics before and after the respective CCM. As previously discussed for the 10 μA measurement, the characteristic curve after the CCM is usually shifted to higher voltages, due to the degradation of the emitter caused by the constant current load. Furthermore, related pairs of characteristics generally move towards higher extraction voltages with each step of the CDLT due to the increasing total current load. Note that the first two current steps of this measurement are an exception, as the 10 nA (black) and 100 nA (dark purple) measurements exhibit a widely scattered and partially non-linear behavior and start at higher voltages compared to subsequent measurements performed at higher set currents. These effects are a result of switch-on and conditioning effects mentioned earlier. Apart from this, the respective characteristic curves after each CCM are typically almost congruent with the characteristic curves prior the subsequent CCM. This indicates, that the most significant changes occur between the pre- and post-curves of a given current level. Consequently, the majority of the current-dependent degradation takes place during the CCM rather than during the sweeps.

The degree of degradation as a function of operating time during the CCM is illustrated in Figure 4.8(b) using the extraction voltage required to obtain the set emission current. While there was a noteworthy initial rise in extraction voltage throughout the initial 10 nA measurement, current levels up to 4 μA and below displayed a minimal voltage increase during the CCM. Here, only a parallel offset attributed to the higher current level can be perceived. For higher currents, the slope of the curves gradually increases until the measurement stops at a

current level of 100 μA after approximately 8 minutes due to the extraction voltage maximum of 1.4 kV being reached. It is evident that the curves mostly follow a linear path. Hence, the linear fit of this data can be used to quantify the current-dependent degradation during the CCM. The y-axis intercept of this fit quantifies the shift of the curves towards higher voltages due to the increasing set current, while the slope can be used to express the mean degradation of the FEA over time resulting from the constant current load. However, as previously mentioned, relating the measurement data to the extraction voltage can be disadvantageous for comparability, especially when measuring in diode configuration. To overcome this issue, the slope and y-axis intercept can be divided by the sample specific distance between extractor and emitter apex. This way, these values refer to the respective applied electric field instead of the extraction voltage, thus enabling a fair comparison among various types of FEAs. Furthermore, to make representative statements about entire types of FEAs, statistical analysis is necessary. Thus, in this work the CDLT is typically conducted on at least three samples of the same type, unless otherwise specified.

Based on the statistical analysis of the fitted data, further insights can be gained from the CDLT. Figure 4.9(a) shows the correlation between the initially required electric field, obtained from the y-axis intercepts as shown in Figure 4.8(b), and the respective set current level for a total of three measured TMAH FEA samples. Additionally, the graph includes an inset displaying the data in adapted ML coordinates. Figure 4.9(b) illustrates the mean electric field shift during the CCM, obtained from the slopes as shown in Figure 4.8(b), for each set current level. The individual sample data is plotted in both graphs as blue, yellow, and green dots. The red line with cross markers corresponds to the averaged result of all three measurements. Although the data points of the individual FEAs are relatively widely scattered, there is a clear trend in the general course. *Edler* concluded in his thesis that this trend can be sufficiently reconstructed by averaging a statistical set of three emitters [191]. The maximum current level may vary between the CDLTs of the individual FEAs. To avoid distorting the mean results by averaging too few emitters, current levels are only assessed if at least two of three FEAs achieve them.

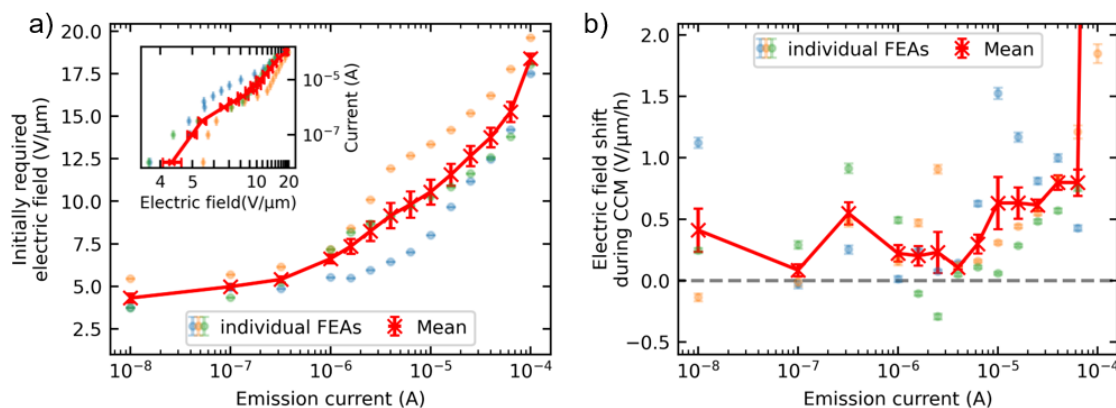


Figure 4.9 Statistical evaluation of the CDLT results for a total of three TMAH FEA samples. The data of the individual samples is plotted in blue, yellow, and green. The red graphs correspond to the averaged results of all three samples. In (a) the initially required electric field to emit the respective set current level of each CCM is plotted. The inset depicts the same data in adapted ML coordinates. (b) represents the change of the electric field over the course of each one-hour CCM.

Based on the inset in Figure 4.9(a), it is evident that the initially required electric field increases mostly linearly with the set emission current when plotted in ML coordinates. Therefore, the increase can be at least partly attributed to the rising current level, as it follows FN-behavior. However, it is also affected by the accumulating degradation of the FEA during the sweeps and the CCM of each set current level, which is probably the cause for the changing slopes. The two effects cannot be differentiated in either the normal or ML coordinate representation. Therefore, this figure serves primarily to compare the durability of the respective emitters during prolonged lifetime testing. An isolated impression of the degradation can be obtained from Figure 4.9(b). It shows emitter-type specific field shift maxima, indicating current levels with significant degradation. For the TMAH FEAs depicted herein, a consistently elevated degradation is observable during the CCM for emission currents beyond 10 μA , for example. Notably, all three samples experience excessive deterioration at a CCM current of 100 μA , which ultimately led to the termination of the CCM and the entire measurement.

Apart from this, the current-dependent degradation can also be estimated using the field enhancement factor, since it is correlated to the radius of curvature, height and surface occupancy of emitting tips [33], [51], [53], [83], [96]. However, as discussed in Chapter 3.1, the FEF and effective emission area must be interpreted with caution. Particularly at the high chamber pressures of 10⁻⁵ mbar found here, adsorbates may cause significant, reversible deterioration of the FEF. This cannot be distinguished from irreversible degradation caused by morphological alterations in the emitting tips. Furthermore, the provided measurements represent entire arrays of simultaneously emitting tips. Consequently, the FEF and associated

effective area are typically a superposition of many individual non-ideal field emission systems [52], [98]. Nevertheless, the relative alteration of these parameters could provide valuable insights into the degradation processes occurring during CCM, if they are interpreted correctly. As described in Chapter 2.4, they can be calculated using the linear fits of the characteristics before and after each CCM. The data resulting from these evaluations is presented in Figure 4.10. Here, only the mean values obtained from averaging all three datasets of the individual emitters are plotted. The solid lines illustrate the temporal course of the respective parameter. The opaque data points with the left arrow belong to the characteristic before CCM, while the semi-transparent data points with right arrow belong to the characteristic after CCM.

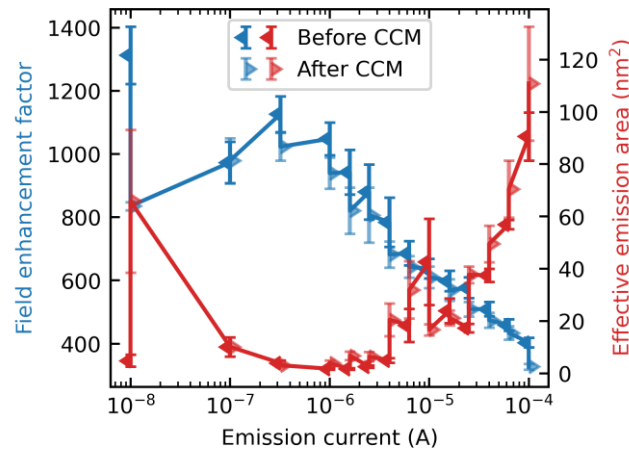


Figure 4.10 Statistical evaluation of the current dependent change of the field enhancement factor and effective emission area for three TMAH FEA samples. The plot shows the averaged data of all three emitters before and after each CCM.

Note the significant inverse correlation between effective emission area and FEF. Due to the questionable physical interpretation of this area-like parameter [33], [195], the discussions in this and the following chapters will be focusing primarily on the analysis of the FEF to avoid redundancy. As expected, the FEF decreases constantly with increasing set current after the first two current levels, where the conditioning is still present, due to the increasing degradation with the current load. Similar to the field shift considered above, the most significant degradation typically occurs during the respective CCM. An improvement may even be observed during the transition from FEF after to FEF before the CCM at the next current level. This might be attributed to the additional activation of new tips that results from the generally increasing extraction voltage or electric field required to reach the higher currents. Notably, set currents levels above 40 μA also cause significant degradation during sweeps, as these currents appear to force individual tips to emit at excessive current densities. Even though the final CCM at 100 μA lasted only 8 minutes, there is still a significant degradation

of the FEF. A possible explanation is that at this stage, the majority of the sharp emitters failed, which ultimately led to the failure of the FEA.

5 Field Emitter Arrays for Diode Configuration Measurements

As stated in Chapter 1, the objective of this dissertation is to develop a silicon-based field emission electron source with an integrated extractor for high-current applications. Prior to the integration of the extractor electrode, a suitable emitter morphology must be established that is able to sustain high current densities reliably in adverse conditions. The work is based on the so-called TMAH FEAs, which have been developed and thoroughly analyzed in prior research [3], [191], [196]–[198]. Here, they serve primarily as a reference and to characterize the newly developed measurement method introduced in Chapter 7. For this reason, their fabrication process is only briefly presented in the following subchapter. A detailed characterization of this type of FEA can be found in [3] and [191]. The silicon nanowire emitters presented in the subsequent subchapter represent a novel approach, which will be discussed in more detail. A comparison of the emission performance of both FEA types in diode configuration using the CDLT will determine their potential to fulfill the necessary requirements for a portable X-ray device.

5.1 Wet-Chemically Etched Field Emitter Arrays

The purpose of this FEA design was to develop a versatile field emission array for easy prototyping that does not rely on photolithography or other expensive clean room methods to produce. Therefore, the number of tips in the array and the dimensions of the FEA should be effortless to adjust for diverse applications and tests without the need for expensive and time-consuming design and manufacturing of photolithography masks. Figure 5.1 schematically illustrates the fabrication process for the TMAH FEAs.

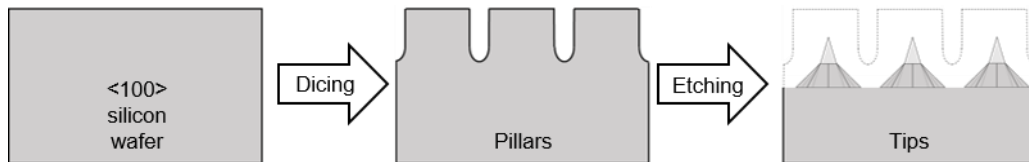


Figure 5.1 Schematic cross-section diagram of the key processing steps for TMAH field emitter array fabrication.

The process starts with pre-patterning mesa structures on a <100> silicon wafer using a wafer dicer. Typically, these structures are pillars with square bases, aligned parallel to the {100} planes, and ranging in height from tens to a few hundred micrometers. Next, a wet

chemical cleaning procedure is performed, followed by anisotropic etching of the silicon structures TMAH. As detailed in Chapter 4.1.1, TMAH exhibits etch rates in silicon that are dependent on crystal orientation. The etch rate ratios of different planes are greatly influenced by both TMAH temperature and concentration [169]. Our previous research demonstrated that by appropriately choosing the TMAH concentration, temperature, and etch time, the etching profile can be tuned to form sharp pyramidal tips on a hexadecagonal basis [3], [196]–[198]. The upper tip is thereby formed by underetching of the convex pillar corners through the fast-etching $\{103\}$ planes, while the lower formally concave cornered base is dominated by the slower-etching $\{221\}$ and $\{331\}$ planes. The spacing between individual emitters, the size, and the embedding depth of the tip relative to the chip surface can hereby be controlled by adjusting the dicing parameters. By altering the shape of the mesa structures to be etched, linear or sheet-like emitter structures can also be realized [192].

The types of TMAH FEAs produced and measured in this dissertation differ only in the number of emitter tips and the active area covered by the array. For the electrical characterization in this chapter, FEAs with an active area of 4 mm x 4 mm are chosen, analogous to the active area of the silicon nanowire FEAs presented in the following subchapter. A $\langle 100 \rangle$ silicon wafer with a low n-doping (phosphorus, resistivity of 10–20 Ωcm) is used as the substrate. Doping concentrations of this magnitude are often used in literature in order to achieve a balance between high substrate conductivity, typically resulting in a high emission currents, and localized self-limitation, which enhances emission uniformity [143]–[145]. The mechanical pre-patterning process with the wafer dicer imposes limitations on the packing density of tips feasible in this process, since this it tends to break off the desired square-based pillars if the pitch is too small. Empirical testing has determined that an array of 60 x 60 tips with a pitch of 66.7 μm can be reliably achieved, leading to its selection. This pitch dictates the height of the etched tips, which is approximately 30 μm in this case. The embedding depth of the emitter is determined by the height of the pre-structured pillars, but care must be taken not to choose this too low, as this would fail to create the desired pyramidal structures. The intended height of the pillars, which is 35 μm , is determined by the blade height of the wafer dicer. However, since this value is usually subject to large variations, the actual embedding depth of each FEA is determined in a scanning electron microscope (SEM) at the end of the fabrication process. After pre-structuring, the samples undergo wet-chemical cleaning using the RCA standard clean process [199]. Subsequently, the samples are immersed in 1% hydrofluoric acid for 60 seconds, which effectively removes both the silicon's natural surface oxide and the surface oxide created during cleaning. This step positively impacts the process

reproducibility. Finally, the tips are patterned in a 20% TMAH solution at 80 °C for approximately 25 to 35 minutes. The etching duration can differ significantly due to coarse nature of the pre-patterning process with the wafer dicer. Furthermore, the doping level of the wafer may impact the etch rate [166]. Therefore, the etching process is conducted by sight. A detailed process sheet for the fabrication of TMAH FEAs with all relevant parameters can be found in Table 2 in Appendix A. Figure 5.2 displays scanning electron microscope (SEM) images in different magnifications of a typical TMAH FEA produced using this process.

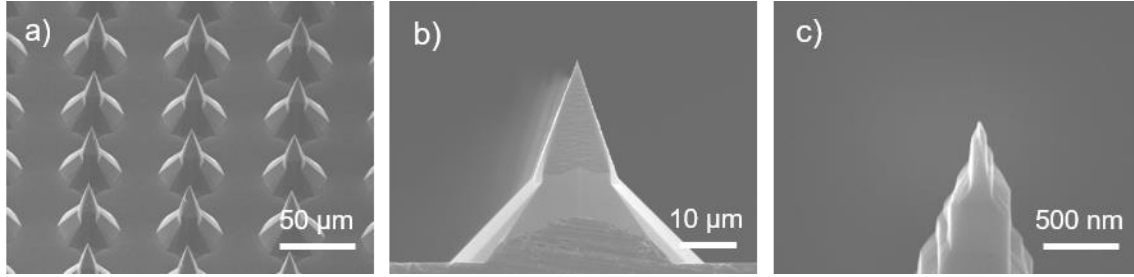


Figure 5.2 SEM images of (a) part of the array, (b) a single tip in sideview and (c) a high magnification image of the tip's apex of a TMAH FEA.

Edler et al. were able to show by geometric SEM analysis of the tip radius distribution that an average tip radius of 11 nm is typically obtained for this type of FEA [3]. To examine the operating range of the TMAH FEAs, three emitters were measured using the current-controlled IV characterization described in Chapter 4.3. Four averaged characteristic curves were recorded for each emitter, with successive maximum set current levels of 1 μA , 10 μA , 100 μA , and 1 mA, at a chamber pressure of 10^{-5} mbar. Each characteristic curve measurement consisted of six individual sweeps. The distance from the extractor to the tip was 80 μm in this instance, resulting in a maximum electric field of 17.5 V/ μm for the maximum extraction voltage of 1.4 kV. Figure 5.3 depicts the performance of the best FEA out of the three.

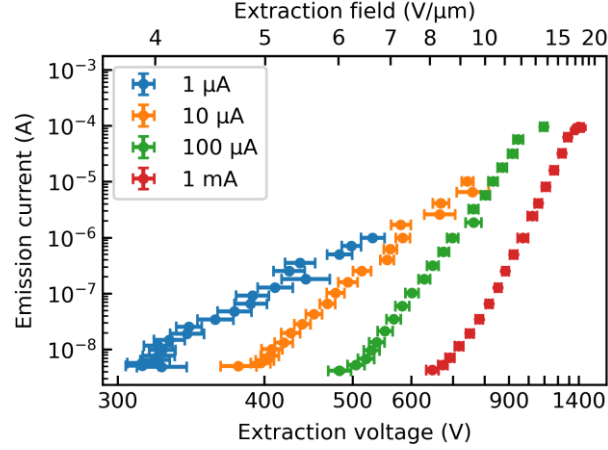


Figure 5.3 Current controlled IV characteristics for one of three measured TMAH FEAs. The data is plotted in adapted ML coordinates with non-reciprocal voltages. A total of four different maximum set current levels were measured with six sweeps each.

Significant degradation can be seen in the voltage shift between the curves. The 100 μA data point on the green characteristic displays noteworthy deviation from the rest of the curve, which indicates exceeding degradation for currents in this range and above. In the subsequent 1 mA measurement, the FEA shown reached the 1.4 kV extraction voltage limit, corresponding to an electric field of 17.5 V/ μm . However, the emission current could not surpass 100 μA . The two other FEAs that were measured, but not shown here, even failed to achieve 100 μA . Consequently, this type of FEA can be considered unsuitable to provide the targeted emission current level of 100 μA to 10 mA over relevant lifetimes in this configuration. However, the emitter packing density of this FEA type is limited by the wafer dicer fabrication process and the extractor-to-tip distance is relatively high, leading to low electric fields. Therefore, an altered fabrication process that allows a smaller lateral spacing and an integrated gate might increase the maximum emission current. Nonetheless, all TMAH FEAs measured in this study showed increased degradation for emission currents of 100 μA and above, which could be attributed to the overall morphology of this type.

5.2 Silicon Nanowire Field Emitter Arrays

In general, FEA failure is often caused by thermal effects due to excessive current densities. High current densities typically result in the melting and evaporation of the emitting material [3], [200], [201]. Consequently, the pressure in the immediate vicinity of the tip increases. The elevated pressure subsequently amplifies other degradation mechanisms such as ion bombardment [202], [203] or surface contamination with adsorbates [3], [83], [204], [205]. In addition, *Kyritsakis et al.* demonstrated with a multi-scale atomistic simulation of copper nanotips, that field-induced forces acting on the partially molten emitter can elongate

and sharpen the tip, resulting in a positive feedback thermal runaway [206]. Ultimately, this can lead to vacuum arcing and/or thermal explosion of the emitting structures [116], [200], [201], [203], [204], [206]–[209]. Thermally induced melting of individual tips was also found to be a common cause of irreversible degradation for the TMAH FEAs described above [3]. To counteract this, the local current density can be limited by restricting the emission current of individual emission sites. As discussed in Chapter 3.2.3, several studies suggest, that the incorporation of nanowires and/or nanochannels as a ballasting element in the current path of individual emitters can effectively restrict the local emission current, leading to a more even current distribution across the array [112], [143]–[145], [148], [210], [211]. Even though there are differing explanations and models regarding the origin of the current limitation, this approach holds promise for enhancing the lifespan of FEAs. Additionally, vertical silicon nanowires possess a naturally high aspect ratio, leading to significant improvement in field enhancement. Their nanometric footprint also permits high packing densities of individual emitters, making them ideally suited for high-current FEA applications.

Fabrication processes for vertical silicon nanowires typically require advanced lithography tools and/or perfectly adapted multilayer processes using state-of-the-art equipment [161], [212], [213]. In contrast to that, the manufacturing process of the vertical SiNW FEAs presented here is based on a simple approach, using a well-known defect of anisotropic reactive ion etching to create needle-like structures with nanometric diameters at the edge of pre-patterned mesa structures without relying on any lithography. To illustrate the concept, a schematic process flow is presented in Figure 5.4. The fabrication primarily relies on the Bosch process, a well-established method for DRIE of silicon with extensive use in the semiconductor industry for many years [166], [175], [214]. Typically, this anisotropic plasma-based dry etching technique involves cyclic repetition of a sequence consisting of isotropic deposition of a passivation layer, followed by a combination of anisotropic physical and isotropic chemical ablation. Through proper parameter selection, precise vertical etch profiles can be created by physical etching of the passivation on horizontal surfaces through directional ion bombardment. For a comprehensive description of RIE etching and the Bosch process, please refer to Chapter 4.1.2. A detailed process sheet for the fabrication of the SiNW FEAs measured in this work can be found in Appendix A, Table 3.

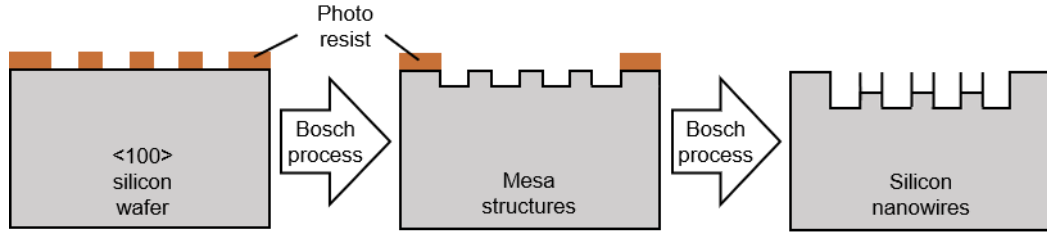


Figure 5.4 Schematic cross-section diagram of the key processing steps for silicon nanowire field emitter array fabrications.

In the first step of the SiNW FEA fabrication, mesas with vertical sidewalls are formed on the wafer surface using photolithography and the Bosch process. Subsequently, a second lithography is applied where only the edge areas of the FEA die are coated with photoresist, leaving the mesas unprotected. This second lithography step is optional since it does not affect the formation of nanowires in the active area of the FEA. Its purpose is to avoid etching of the surrounding die area, simplifying sample handling by preserving pristine surfaces along the sample edges. The crucial part of the fabrication is the non-optional second Bosch process. During the initial passivation step of this DRIE process, the passivation layer conformally covers all surfaces. In the subsequent etching step, complete physical removal of the passivation layer at the vertical mesa edge is unlikely due to the low probability of ion impact. Consequently, the remaining passivation protects the underlying silicon from complete chemical erosion. After multiple cycles, this self-masking process ideally yields needle-shaped silicon nanowires on the edge of the mesa structures. The decisive factor for the nanowire formation is achieving the most vertical etch profile possible in both DRIE processes. To obtain such a profile, system-specific optimum of numerous multilaterally linked process parameters must be found, which will not be discussed in detail here, as this would go beyond the scope of this work. However, in a successfully optimized process, the density of the nanowires can be controlled predominantly by the ratio of etch and passivation duration, while the number of cycles mainly determines their height. Figure 5.5 displays SEM images of a typical outcome of this two-step nanowire fabrication procedure. As can be seen in Figure 5.5(b,c), the nanowires lengths are typically in the range of a few microns. Their diameters may vary from a few nanometers up to microns, depending on the specific process parameters and mask layout. In this work, all fabricated nanowire structures display a sequence of nano-constrictions along their length, visible in the magnified inset in Figure 5.5(c). These result from the Bosch process and have the potential to act as nanochannel-elements to further restrict emission currents of individual emitters in the array [112], [148].

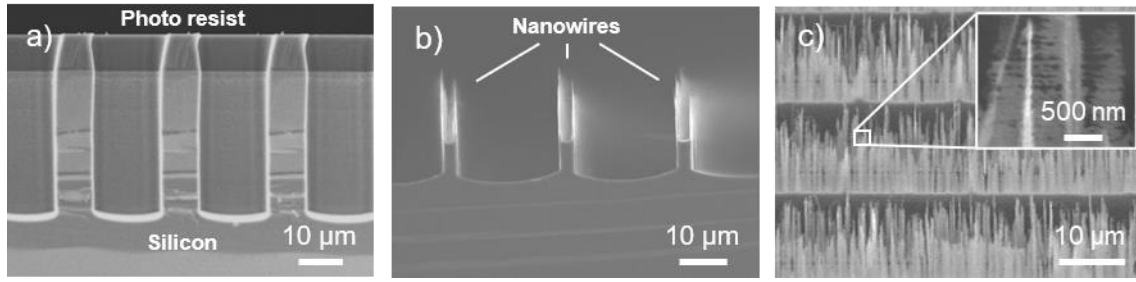


Figure 5.5 SEM images of (a) the mesa structures before the silicon nanowire etch and of the silicon nanowires after etching in (b) cross-section and (c) oblique top view including a magnified image of a single nanowire as an inset.

In a recent proof of concept publication, we demonstrated that this type of FEA can emit currents of up to 20 mA during IV sweeps with minimal degradation at an electric field of 26 V/μm and a chamber base pressure of 10^{-7} mbar [215]. It should be noted that the maximum current in this case was limited by the current capacity of the setup's power supply, as the extraction voltage threshold of 1.4 kV was not reached during the measurement. Considering the FEA's active area of 0.16 cm², the resulting current density is approximately 0.1 A/cm². Therefore, these results fall short to the latest state-of-the-art maximum values by three to four orders of magnitude (see Chapter 3.2). However, a direct comparison of these values is not feasible as this measurement's operating pressure was higher than that of the literature, the power supply limitations restricted the maximum current, and the FEAs studied in the literature had an integrated gate, leading to increased extraction fields due to the shorter distances between the emitter and extractor. Furthermore, this type of FEA offers substantial potential for optimizing the emitter density. For comparison, the lateral spacing between individual emitters in *Guerrera's* study [160] was 1 μm in each direction, whereas in this study, the line-shaped mesas had a spacing of either 25 μm or 50 μm with mesa diameters of 5 μm and 7.5 μm.

To facilitate the understanding of the geometry used, Figure 5.6(a) displays a top-view diagram of the emitter die, featuring the mesa and active area dimensions. Here, the white areas represent the etched pits resulting from the DRIE process, while the gray areas indicate the masked silicon surfaces during the first Bosch process. Note that the edges of the mesas merge into the edge area of the chip and are not self-contained, since the lithography mask used in this study was originally intended for a different purpose. Consequently, this layout is unsuitable for future gate integration because it does not allow the fabrication of symmetrical gate apertures surrounding the nanowires located on the mesa edges. Thus, a new lithography mask was designed with mesa structures that have enclosed edges. The mask design examines three different mesa geometries: two circular and one line-shaped. Top-view diagrams of the different FEA designs are shown in Figure 5.6(b). The only difference between

the two circle-shaped designs is the diameter of the mesa with 5 μm or 10 μm . In this work, they will be referred to as CD5 and CD10, where the numbers represent the respective diameter of the mesa in microns, and the letter “C” represents the circular shape. Accordingly, the line-shaped mesa, which has a 5 μm diameter, is called LD5. The active area of each layout is 4 mm x 4 mm, and the lateral spacing between the mesas is always 40 μm . Note that this mask is one of four in a set used for the multistep gate integration, detailed in Chapter 6. For the initial test, the dimensions were conservatively selected to avoid exceeding the alignment accuracy limit of the mask aligner.

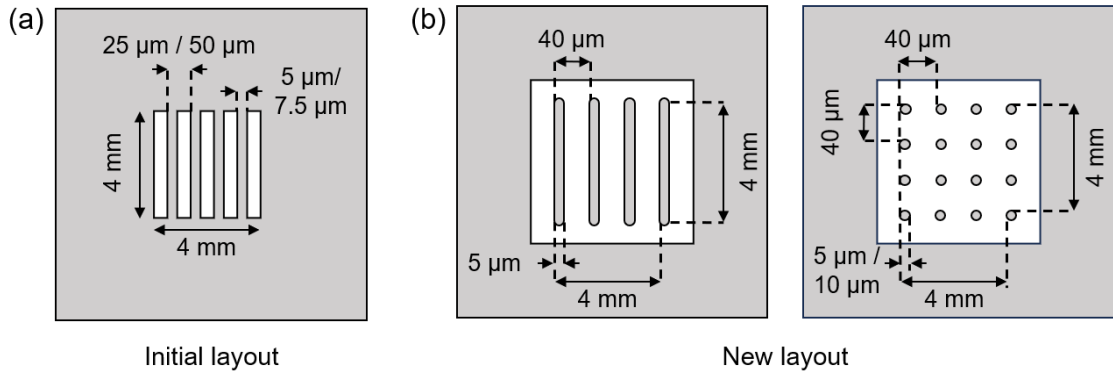


Figure 5.6 Schematic layouts of the different SiNW FEAs for diode configuration measurements. The design in (a) was used for the initial proof of concept study [215]. (b) is the updated design intended for the integration of an extraction gate. Three different FEA geometries are available in the new design: one with line-shaped mesas, and two with circular mesas which differ only in mesa diameter.

To obtain a first impression of the operating range of the SiNW FEAs fabricated using this mask, three samples of each SiNW FEA type were evaluated at a measurement pressure of 10^{-5} mbar using current controlled IV characteristics. Consistent with the previous investigations of the TMAH FEAs, low n-doped (phosphorus, resistivity of 10–20 Ωcm) $\langle 100 \rangle$ silicon wafers were used as the substrate material and set currents up to 1 μA , 10 μA , 100 μA and 1 mA were measured. Six individual sweeps were conducted for each sub-measurement. Figure 5.7 displays an SEM image and the measurement results for one sample of each emitter type. The SEM images also include insets featuring enlarged views of each emitter structure investigated in this study along with its corresponding name. In these measurements, the separation between the extractor and the tip was 70 μm because the SiNW FEAs's embedding depth is ten microns less than that of the TMAH FEAs. To ensure a fair comparison, the TMAH FEAs' maximum electrical field of 17.5 V/ μm is denoted as a gray dashed line on the measurement charts in Figure 5.7(b,d,f). Note, that the height, shape and density of the nanowires shown in Figure 5.7(a,c,e) differ from those depicted in Figure 5.5 despite the unchanged process parameters. These variations are due to a well-known phenomenon in RIE,

often referred to as ‘loading effect’ [216]. It describes the dependence of the etch rate on the ratio of exposed to masked silicon surface as defined by the mask design, due to the limited local density of reactants available. higher silicon surface exposure typically leads to a decreasing chemical etch rate. Accordingly, the etch rate and profile of DRIE process are typically affected by changing the mask layout [174], [216], [217]. Since the mask contains different types of SiNW FEAs with varying coverage ratios, a DRIE parameter optimization study would be needed for each individual structure to achieve optimal results. However, at this stage of the FEA development, there is potential for significant changes in the design and the optimal nanowire shape has yet to be found. Therefore, the etch parameters that were optimized for the previous proof of concept design were not altered for these studies. Additionally, it should be noted that the herein used RIE system is only equipped with a two-inch plasma source, which can result in inhomogeneous etching outcomes over the wafer area with radial dependence while etching four-inch wafers. Consequently, there may be differences in morphology between chips of the same type.

Based on the SEM image in Figure 5.7(a), it is evident that the non-optimized process parameters resulted in substantial under-etching of the mesas for the CD5 FEA. In some regions, several pillars even broke off. Consequently, all three samples tested utilizing this design exhibit the weakest performance among the SiNW FEAs studied, and none of the samples achieved the final maximum set current of 1 mA (see Figure 5.7(b)). In contrast, the measurement of the CD10 FEA, depicted in Figure 5.7(d), shows a significantly better performance compared to the CD5 or the TMAH FEA (Figure 5.3). The larger mesa diameter resulted in the preservation of all structures, enabling all three measured FEAs to reach the maximum current of 1 mA within the range of the reference electric field of $17.5 \text{ V}/\mu\text{m}$. Therefore, the CD5 samples exhibit a maximum emission current that is 10 times higher than the best performing TMAH sample at a similar electric field. Noteworthy is the minimal degradation observed in the initial three current levels of Figure 5.7(d). Throughout the course of the measurements conducted at $1 \mu\text{A}$, $10 \mu\text{A}$, and $100 \mu\text{A}$, the extraction voltage necessary to achieve the desired current levels remained virtually constant. During the 1 mA measurement a slight shift in the characteristic towards higher currents is noticeable. However, upon closer examination of the individual sweep data, it becomes apparent that this shift only occurred during the first sweep. The characteristic remained stable in subsequent sweeps, as indicated by the small error bars on the x-axis for the averaged characteristic. The measurement of the LD5 FEAs show similar results. Once again, all three emitters were capable of achieving the desired current of 1 mA with less than 1.4 kV of extraction voltage. For the

depicted measurement in Figure 5.7(f), the characteristics even shifted towards lower extraction voltages with each sub-measurement. This suggests that this type of FEA can resist high currents up to 1 mA without significant degradation even at an elevated pressure level of 10^{-5} mbar.

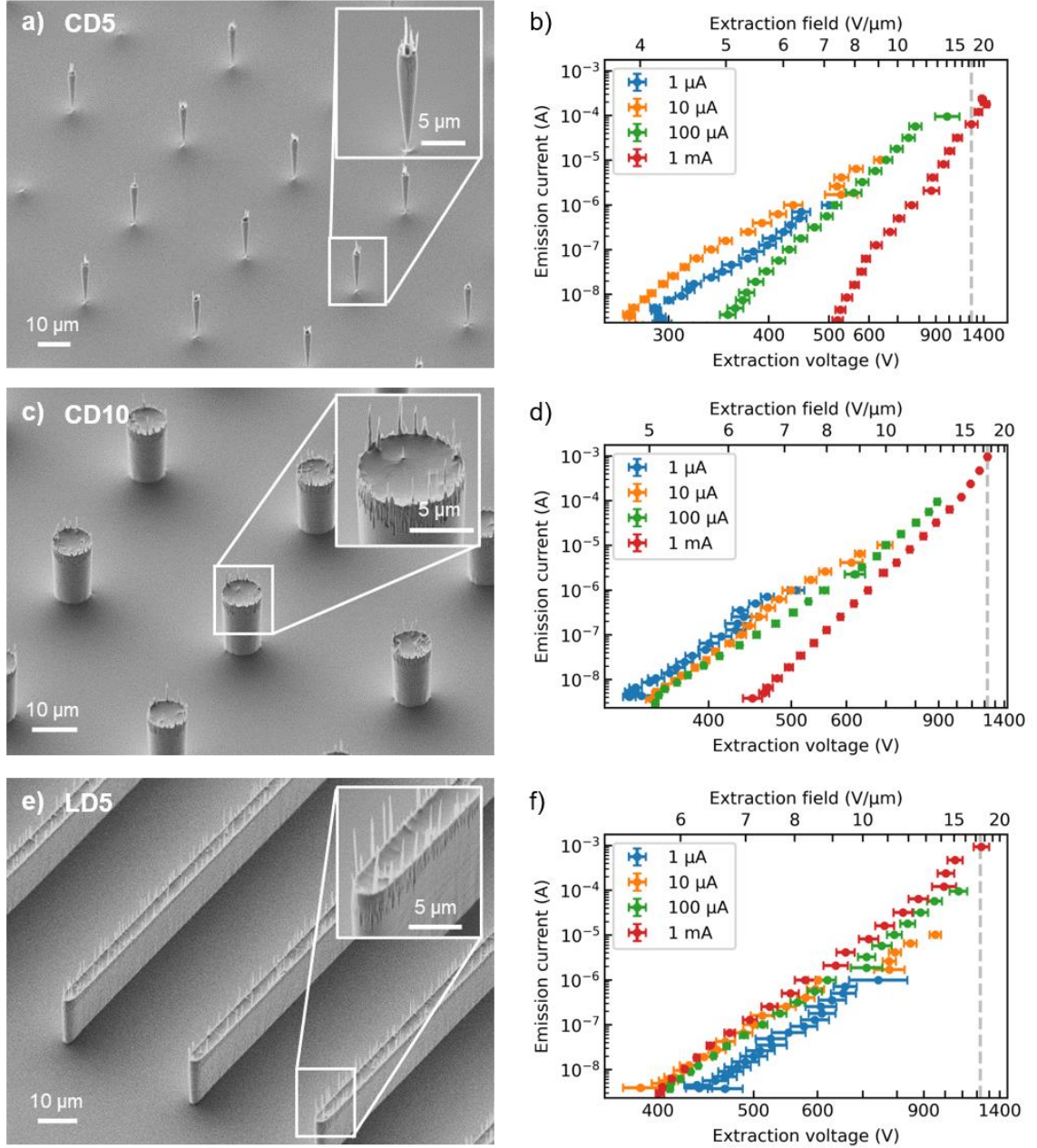


Figure 5.7 (a,c,e) SEM images of the three different types of SiNW FEAs. (b,d,f) show the corresponding current controlled IV characteristics in modified ML-coordinates measured at a pressure of 10^{-5} mbar. The emitting structures of all types are approximately equal in height but differ in the layout of the mesa. The dashed line indicates the maximum electric field achieved by the TMAH FEAs for reference purposes.

5.3 Comparative Characterization

To further investigate the fabricated FEAs, this study compares their current-dependent performance using the CDLT outlined in Chapter 4.2.4. First, the various types of SiNW FEAs are compared to the standard TMAH FEAs. Subsequently, a comparative analysis of morphologically similar SiNW FEAs with different doping is performed to identify opportunities for optimizing the local current limitation of the nanowires. All measurements are conducted in diode configuration at a base pressure of 10^{-5} mbar.

5.3.1 Comparison of Different Field Emitter Array Types

For the first study, three samples were prepared for each type of emitter. Low n-doped (phosphorus) $\langle 100 \rangle$ silicon wafers with a resistivity of 10–20 Ωcm were used as the substrate material. To account for potential morphological variations, the SiNW FEA samples were chosen from different positions on the wafer. Figure 5.8 presents the averaged results obtained from the CDLT for each emitter type regarding the initially required electric field per set current level and the degradation during the corresponding CCM. Note that, as mentioned in Chapter 4.2.4, only current levels reached by at least two of the three measured FEAs per type are evaluated in the averaged results (solid line). The maximum currents achieved by the individual samples vary significantly in this measurement. For this reason, semi-transparent stars in the respective emitter color are included in the graph to depict the final data points of each individual sample.

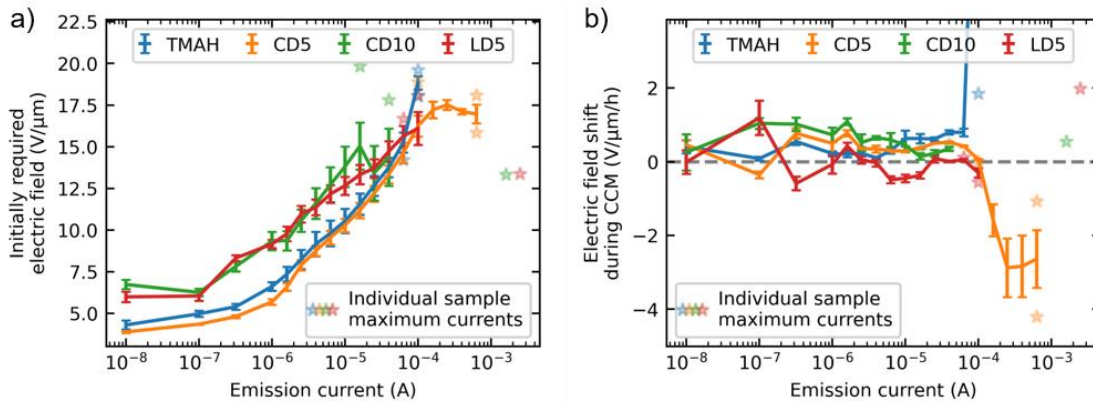


Figure 5.8 CDLT results for the various types of FEAs fabricated. For each type, three samples were evaluated. The averaged results (solid line) only comprise data points attained by at least two of the three samples. The star markers indicate the highest current achieved by each sample for each type.

The course of the initial required electric field in Figure 5.8(a) shows a relatively similar pattern for all measured samples. On average, the CD5 FEA achieved the highest currents, with two out of three emitters capable of emitting up to $630 \mu\text{A}$ for the full hour of CCM.

The CDLTs terminated as they could not reach 1 mA during the sweeps and CCM of the subsequent sub-measurement. In the case of the CD10 and LD5 FEAs, one sample each survived up to 1.6 mA and 2.5 mA respectively. Due to the one-hour CCM per current level, this corresponds to a total operating time of 7 and 8 hours respectively at emission currents beyond 100 μA and a pressure of 10^{-5} mbar. On average, however, these two types of emitters only achieved a maximum of 100 μA and 40 μA , respectively. All three TMAH FEAs reached a maximum current level of 100 μA . The CD5 and TMAH emitters initially require lower field strengths, but these increase more rapidly with rising emission current compared to the CD10 and LD5 FEAs. As a result, all curves converge in the upper 10 μA range. A variation can be observed in Figure 5.8(b), which displays the degradation in electric field shift throughout the CCM. All graphs show significant fluctuations, mostly between 0 and 1 V/ $\mu\text{m}/\text{h}$. However, only the CD5 and LD5 emitters demonstrate negative degradation at multiple current levels. This means that the extraction voltage required to maintain the emission current decreased during the CCM. This phenomenon can be attributed to the conditioning and activation processes in FEAs, as outlined in Chapter 4.2.3. The impact of adsorbates is likely to be predominant due to the relatively high operating pressure. When under constant current, the emitter heats up due to Joule heating and the Nottingham effect [55], [56]. As the temperature increases, the desorption rate also increases, allowing for the reversal of previous adsorbate-induced degradation [83]. In current-controlled measurements, this manifests as a reduction in the extraction voltage or electric field. In case of the TMAH FEA, the constant emission current level of 100 μA caused excessive degradation in all three samples, resulting in premature termination of the CCM and the entire measurement. In accordance with the results from Chapter 5.1, this appears to be the maximum current achievable by this emitter type. Also noteworthy is the averaged behavior exhibited by the CD5 emitters in Figure 5.8(a,b) when the current exceeds 100 μA . The pattern of the curve is very similar to those of the best performing CD10 and LD5 samples. To discuss this in more detail, Figure 5.9(a,b) shows the CDLT results of the best emitters of each SiNW FEA type. In addition, Figure 5.9(c) illustrates the temperature of the sample holder, that was measured during the CCMs of the CDLT. It is important to note that the temperature is measured using an uncalibrated thermistor located on the back of a ceramic plate (see Chapter 4.2.1) and therefore does not directly match the FEA's temperature. Nonetheless, it can be used to monitor the temperature change of the entire sample holder, which correlates with the FEA temperature. Figure 5.9(d) shows the extraction voltage and sample holder temperature over time during the CCM at 1.6 mA of the LD5 sample.

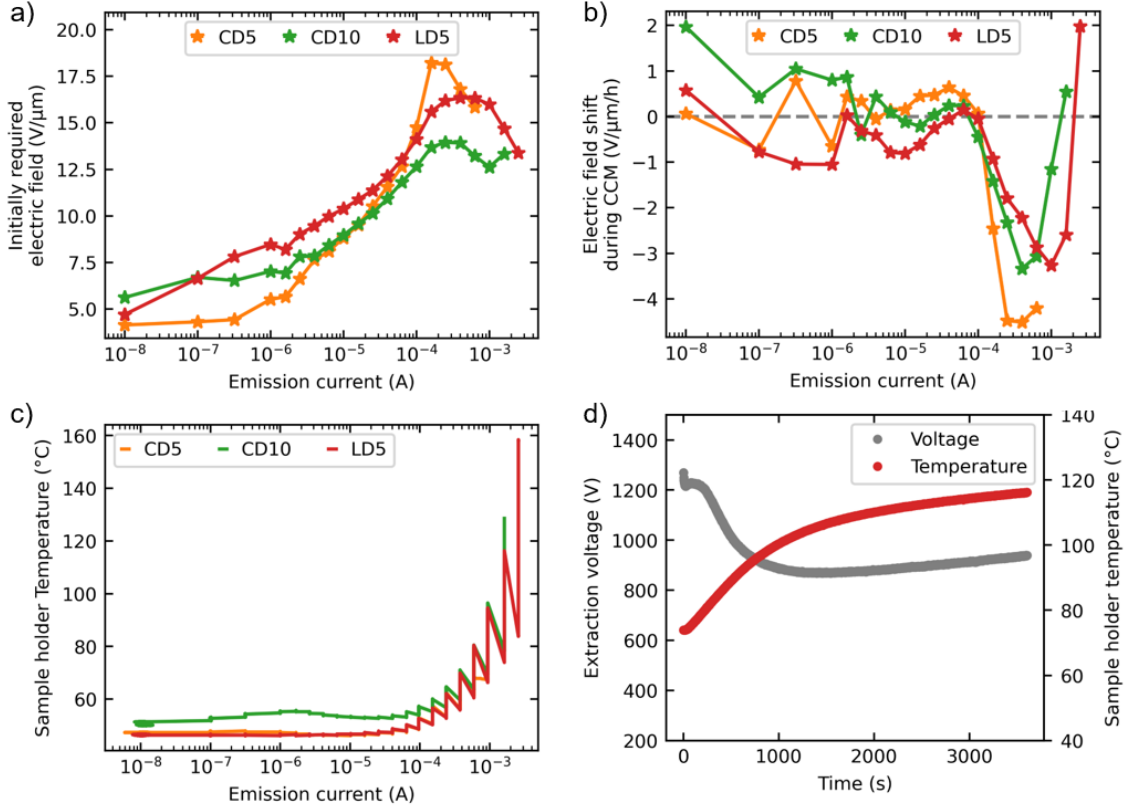


Figure 5.9 (a,b) CDLT results for the best performing sample of each SiNW FEA type. (c) shows the corresponding sample holder temperature measured during the CCMs and (d) the extraction voltage and temperature course during the 1.6 mA CCM of the LD5 sample.

According to Figure 5.9(c), the sample holder temperature increases significantly for emission currents above 100 μA , regardless of the type of FEA sample. The zigzag pattern of the curve indicates that the temperature increase is primarily due to constant current operation. During the sweeps before and after the CCM, the entire system cools down again to some extent. On the one hand, raising FEA temperature can partially recover reversible degradation [3], [83]. On the other hand, it can lead to thermally-assisted field emission [100], [119], [206]. Furthermore, the ballasting effect of nanowires acting as ungated field effect transistors and nanochannels can be reduced by increased temperature [112], [146], [148]. It therefore affects potential current limitation of individual emitters. In any case, the extraction voltage required for a given emission current decreases as a result. This is particularly evident in the temperature and extraction voltage curve shown in Figure 5.9(d). For all samples measured, an inverse relationship between sample holder temperature and extraction voltage can be observed above an emission current of 100 μA . This non-linear behavior distorts the linear fit of the extraction voltage curve in the CDLT and affects the parameters obtained from it. It therefore accounts for the anomalous trend for currents above 100 μA of the initially required electric field and electric field shift during CCM shown in Figure 5.9(a,b). Lower emission currents do not seem to have a significant impact on the FEA temperature. The cause of the

rise in temperature may be self-heating of the emitters due to Joule heat and the Nottingham effect [55], [56]. However, it is unlikely that this generates enough heat in the bulk to be measurable at the thermistor. A more probable reason is heating of the silicon plate, which is used as the extractor in diode configuration measurements. As depicted in Figure 4.4(c), the silicon plate is in direct contact with the entire specimen holder. During measurement, the emitted electrons transfer a large proportion of their energy to the extractor in the form of heat. Extraction voltages in the kilovolt range, coupled with currents up to a few milliamperes, allow for powers in the watt range dissipated on small areas on the silicon plate. The generated heat spreads throughout the entire sample holder including the FEA. This effect will be minimized by a triode setup and a highly transmissive gate structure.

Figure 5.10 depicts the current dependent field enhancement factors and effective emission areas for the measured FEA-types. The general trend of the curves is similar. For emission currents up to $1 \mu\text{A}$ the FEFs and effective emission areas exhibit considerable fluctuations due to conditioning effects and additional tip activation triggered by the rising extraction voltage at each current level. From $1 \mu\text{A}$ upward, the FEF continuously decreases due to degradation that increases with the current load. Above $100 \mu\text{A}$, the previously mentioned thermally induced effects start affecting the CD5 FEAs. The high fluctuations in this current range are likely due to regeneration of reversible degradation. Also, FEA operation outside the MG regime, e.g. by thermally-assisted field emission, can lead to non-linear characteristics and unreasonable fits in this current range [186].

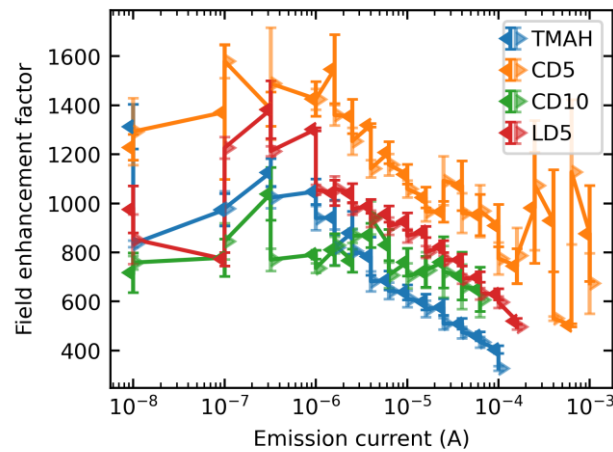


Figure 5.10 Comparison of the averaged current dependent field enhancement factors of each FEA type. The orientation of the marker indicates whether the respective value reflects the FEF before (arrow to the left) or after (arrow to the right).

The difference between FEA-types is primarily found in the absolute values of the respective FEFs. At every current level, CD5 samples show the highest FEFs. The morphology of the emitters likely explains this distinction. The SEM images in Figure 5.7(a,c,e) reveal

that the CD5 FEA typically exhibits a low density of high nanowires on pillars with small diameter, increasing its aspect ratio and field enhancement. On the other hand, the CD10 and LD5 have a density of individual nanowires, which may cause electrostatic shielding of neighboring emitters and thus reduce the FEF [52], [54], [98].

5.3.2 Doping-Induced Current Limitation

To investigate the impact of dopant type and doping level on the emission properties of the FEAs, multiple sets of SiNW FEAs were fabricated using distinctly doped $\langle 100 \rangle$ silicon wafers. Table 1 lists the respective name, dopant, and specific resistivity of the substrates.

Table 1: Name, dopant and specific resistivity of the silicon substrates used for the study.

Designation	Dopant	Specific resistivity (Ωcm)
n	Phosphorous	10–20
n ⁺⁺	Arsenic	< 0.005
p	Boron	10–20
p ⁺⁺	Boron	< 0.005

The CDLT was performed on three LD5 samples of each doping. Figure 5.11 shows the results in form of the initially required electric field to obtain the set current level and the electric field shift during the respective CCM.

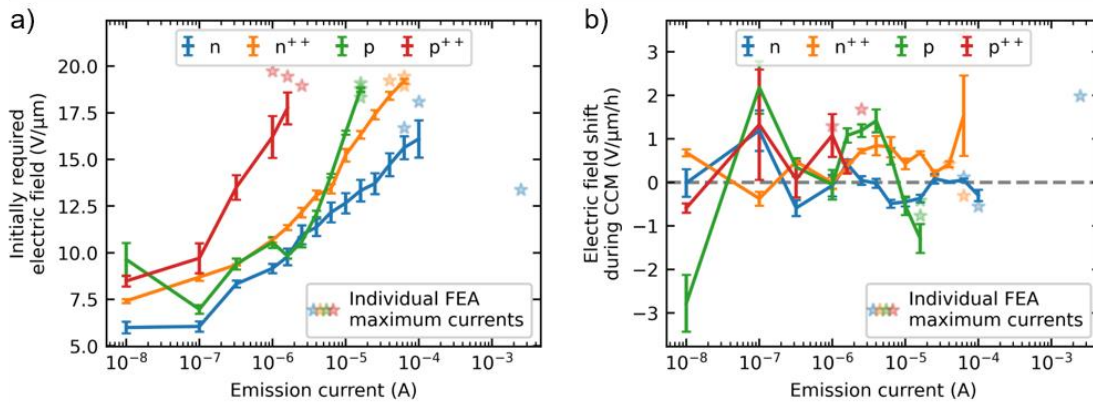


Figure 5.11 CDLT results for differently doped LD5 FEAs. For each doping, three samples were evaluated. The averaged results (solid line) only comprise data points attained by at least two of the three samples. The star markers indicate the highest current achieved by each individual sample for each type.

The already previously demonstrated n-type LD5 FEAs (see Figure 5.8) reached the highest CDLT current of 100 μA for two out of three measured samples, followed by the n⁺⁺-

and p-type FEAs with 63 μA and 16 μA , respectively. The p^{++} -doped emitters only achieved a maximum current of 1.6 μA . Accordingly, the initially required electric fields in Figure 5.11(a) are the highest overall for the p^{++} -type FEA. The graphs for the other types are largely similar. However, there is a notable increase in slope for the p-type for currents exceeding a few microamps. It should be noted that no temperature increase of the sample holder was observed during these measurements, except during the CDLT of the highest performing n-type FEA sample. The electric field shift graphs in Figure 5.11(b) fluctuate strongly for all types. The n^{++} -type exhibits primarily degradation during the CCMs, particularly for currents exceeding 1 μA . This is likely due to its high conductivity, which can reduce the self-limiting effect of ballasting elements on the emission, leading to high local current densities at the expense of emitter lifetime [112], [143]–[145]. For n-type FEAs, the improvement during CCM appears to outweigh the degradation on average, which is the reason for the high maximum currents achieved by this type. It is possible that the lower conductivity and charge carrier density of the nanowires in this case causes effective self-limitation of local emission current, forcing a wider distribution of the current load across the array and thus increasing the FEA's lifetime [143]–[145]. In addition to the potential morphological current limiting of the nanowires, doping-dependent saturation of emission current can be expected for the p- and p^{++} -type FEAs [67], [70], [75], [156]. As outlined in Chapter 2.3, this is due to the formation of a depletion region within the tips during increasing electron emission, which restricts the charge carrier supply of the inversion layer at the apex of the tip. However, evaluating the effectiveness of emission current limitation based on Figure 5.11 is challenging, as the degradation is due to multiple emission current dependent processes occurring simultaneously throughout the entire array. A more isolated insight in the morphology- and doping-dependent limitation and saturation effects can be obtained from the characteristic curves measured during the CDLT. For this reason, Figure 5.12 depicts the mean characteristics taken before the 6.3 μA CCM for each sample of the n-, n^{++} -, and p-type FEAs, along with the 1 μA characteristics before CCM for the p^{++} -doped samples, as this is the maximum current achieved with this doping.

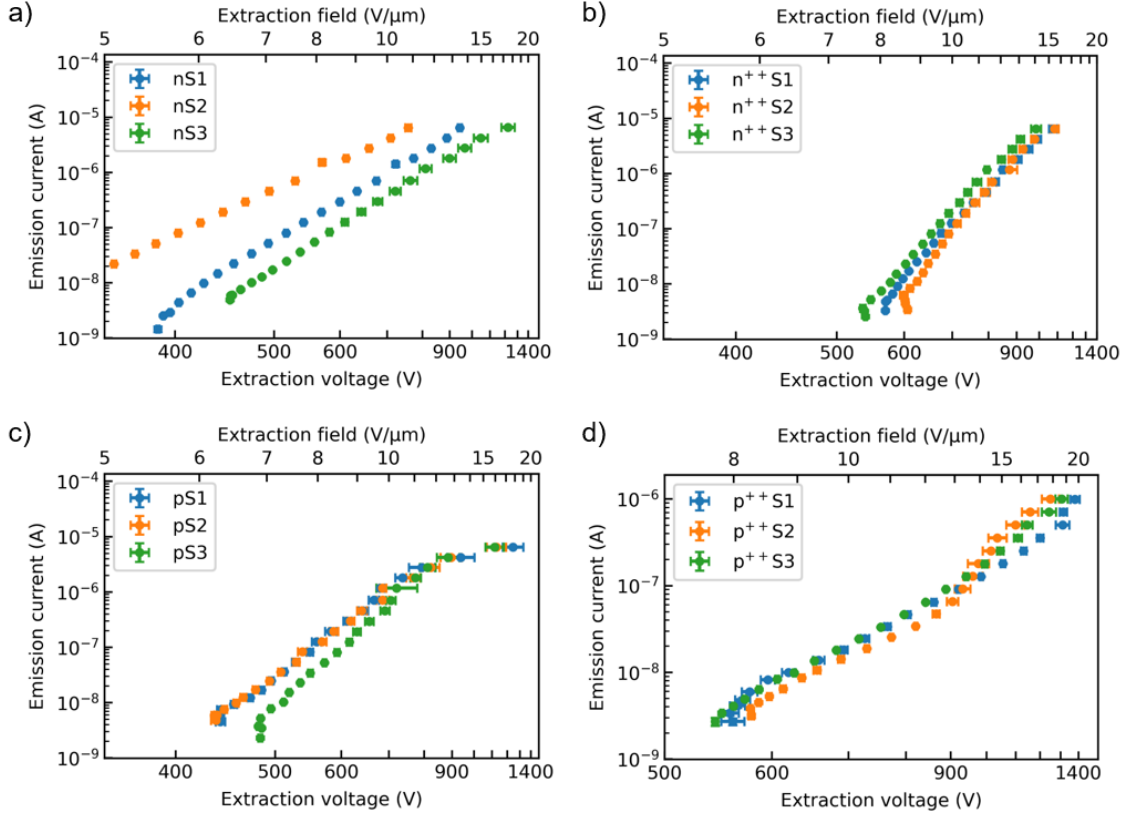


Figure 5.12 Mean characteristics of the three measured (a) n-type, (b) n^{++} -type, and (c) p-type samples before the $6.3 \mu\text{A}$ CCM. (d) shows the p^{++} -type measurements before the $1 \mu\text{A}$ CCM, as this is the maximum current reached by all three samples.

The n-doped and n^{++} -doped FEAs illustrated in Figure 5.12(a,b) exhibit a consistently linear behavior, whereas Figure 5.12(c) reveals a discernable saturation in the upper current region for the p-type samples. This saturation of the emission current becomes apparent in nearly all measured emission characteristics of p-type FEAs for currents exceeding a few microamperes. It also explains the increasing slope of the initially required electric field of this emitter type in Figure 5.11(a), as higher electric fields are needed to increase the emission current in the saturation region. In contrast, the measured characteristics of all n- and n^{++} -doped samples exhibit linear behavior throughout the entire current range. The characteristics of the highly doped p^{++} FEAs, on the other hand, mostly follow a non-ideal linear path, often featuring multiple slopes within a curve, as shown in Figure 5.12(d). However, it is important to note that linear or near linear behavior does not necessarily imply the absence of morphological and/or doping-induced current limitation. The integral characteristics measured in this study always display a superposition of many simultaneously emitting nanowires. The extent and nature of the current limiting effect of each individual nanowire are significantly influenced by morphologic and conditional differences [112], [143]–[145], [148], [190]. As shown in Figure 5.5, there can be considerable variations in the diameter and length of individual

nanowires within an FEA. Moreover, conditional factors such as local temperature, surface occupancy with adsorbates and potentially even the local doping level of the emitters may change during the ongoing operation of the FEAs. Therefore, achieving an ideal saturation behavior, as schematically shown in Figure 2.3, is improbable in integrally measured FEA characteristics that represent potentially thousands of simultaneously emitting nanowires.

The evident and reproducible saturation observed in the p-type characteristic curves suggests that this type of doping is effective in mitigating both morphological and conditional influences. However, the level of doping appears to be a critical factor, as the highly doped p^{++} FEAs fail to display clear saturation behavior. One possible reason for this phenomenon involves the increased field penetration depth associated with lowly doped silicon. In a simulation study conducted by our group, it was demonstrated that the field penetration into p-doped silicon emitters results in an effective change of the electrostatic tip contour and thus to a reduction of the FEF [190]. Deeper field penetration could thus contribute to a superior compensation of geometric inhomogeneities among the emitters in the array. Moreover, the extent of the space charge region formed in the p-doped nanowires during field emission and the impact of surface currents on current limitation may be contributing factors. On the one hand, leakage currents across the surface have the potential to bypass the dopant-induced current limitation, and may fully short-circuit the space charge region in the worst-case scenario. The resistance to leakage currents across the surface increases with the length of the space charge region, which reduces the likelihood of a complete short circuit. On the other hand, surface currents in the form of surface-generated charge carriers are an essential component of the saturation current. Together with charge carrier generation in the depletion region and diffusion current through the depletion region, they define the height of the saturation level. Considering the inherently high surface-to-volume ratio of nanowires, the surface-generated currents are expected to significantly affect the charge carrier supply to the tip in this case. Thus, an increase in the length of the space charge region will result in a considerable increase of the saturation level. Generally, the length of the depletion region in doped semiconductors increases as the dopant concentration decreases [71]. Consequently, the lower dopant concentration of the p-type FEAs leads to a longer space charge region in the nanowires, reducing current limitation bypassing and increasing the current saturation level. Conversely, the highly doped p^{++} FEAs with a shorter depletion region exhibit lower levels of current saturation. Additionally, these saturation levels are concentrated within a narrower voltage range, resulting in a higher probability that the integral characteristic curve simultaneously contains emitters before saturation, in saturation and in avalanche mode. This accounts for the characteristics' shape for this type of emitter. Furthermore, the earlier start of

the avalanche region triggers premature burnout of emitters in the FEA. This explains the generally high and rapidly increasing required extraction fields in Figure 5.11(a) and the early end of the CDLT of this emitter type.

5.4 Conclusion

The RIE-based, self-masking fabrication process of the SiNW FEAs can result in morphological variations among samples of the same type if they originate from different positions on the wafer. As a result, the comparative statistical analysis of TMAH FEAs and various SiNW FEA types using the CDLT did not reveal a clear preference, as the individual sample performances varied accordingly. However, the best performing samples of the SiNW FEAs reached emission current levels of up to 2.5 mA. Although the high emission currents led to self-heating of the entire sample holder including the FEA, which might result in a temperature enhanced performance, this demonstrates the SiNW FEAs' ability to withstand dc currents up to the milliampere range at least for several hours at a base pressure of 10^{-5} mbar. Significantly longer lifetimes can be expected for the minimum requirement of 100 μ A. Additionally, integrating a gate might enhance the lifetime of the FEAs by minimizing the emitter-to-extractor distance to allow for significantly higher extraction fields.

The investigation of the local emission current limitations via doping also reveals promising opportunities for optimization. The highest emission currents were achieved by the n-doped FEAs. However, it is important to note that this does not necessarily mean that this type of doping is superior in every aspect, as this only proves the best performance under the very specific measurement conditions of the CDLT. In particular, the saturation behavior of p-doped samples demonstrates potential to compensate variations in nanowire dimensions, surface conditions and environmental conditions in immediate vicinity of the individual emitters. This could potentially homogenize the current distribution over the array and thereby significantly increase the lifetime of the FEAs at a given current level. The dopant concentration can hereby influence the saturation level and emission stability depending on the specific application and target current. As surface currents are expected to have a major influence on this in the case of SiNWs, passivating the surface (e.g., via thermal oxidation) may result in further stabilization of the saturation level. To optimize the morphologic current limitation imposed by the nanowires, it is possible to homogenize the distribution of their dimensions in the array. A viable approach for this could be an alternative fabrication process that has recently been published by our group [218]. Instead of random self-masking through passivation residues in a maskless Bosch process, the targeted degradation of thin photoresist is used here as an initiator for nanowire formation. The resulting nanowires have uniform height

and diameter, which should lead to a more homogeneous current-limiting behavior across the array [143]–[145]. However, it is evident from the investigations in this chapter that assessing the effectiveness of current-limiting elements in FEAs from integral measurements is only possible to a limited extent, as the overlapping emissions of multiple tips can obscure the outcomes. Thus, a technique is necessary to enable determining emission currents of individual emitters while being operated integrally (see Chapter 7).

6 Field Emitter Arrays with Integrated Gate

After identifying a promising emitter morphology to meet the targeted application requirements in this work, the next step is to integrate an on-chip extraction electrode. To minimize the risk of incorporating known gated FEA vulnerabilities, experience from the literature will be used to develop the concept. This chapter outlines the underlying idea of the process flow, design of the lithography masks and the technological implementation of the gated FEAs developed in this work. The performance of several fabricated samples is then evaluated in triode configuration based on the CDLT methodology.

6.1 Concept Development

As discussed in Chapter 5.2, the most common failure mechanisms of ungated FEAs can be attributed to excessive local current densities. To prevent these, a uniform emission current distribution of the array is necessary, which can be achieved for example through morphological and/or doping-induced local current limiting. For gated FEAs these solution strategies remain relevant, but additional challenges also must be considered. The aim of gate integration is to position the extractor as close and aligned to the emitter as possible, to achieve maximum field strengths at low extraction voltages with high electron transmission to the anode. Unfortunately, the close proximity presents new challenges for the FEA concept. Firstly, it raises the potential for arcing caused by localized pressure increases due to emitter or gate material evaporation or outgassing. Thus, achieving a uniform current distribution to circumvent high emitter current densities and maximizing the electron transmission rate to reduce thermal power dissipation in the gate becomes even more crucial. Secondly, sufficient electrical insulation between the extractor and emitter must be ensured without increasing the distance between them. This is typically achieved by applying one or more insulating layers between the substrate and gate using standard thin film technology processes. To prevent dielectric breakdown of the insulation, it is important to select materials with high dielectric strength and adequate layer thickness. However, the insulator surface adjacent to the vacuum is also a vital weak point of gated FEAs, especially at high pressure levels. Leakage currents across the dielectric surface can significantly reduce the transmission, while increasing the risk of vacuum arcing due to increasing gate current density. Furthermore, it can trigger vacuum surface flashover or dielectric surface breakdown, which are known as critical failure mechanisms of gated FEAs [219]–[222]. Parasitic emissions from the triple point

where vacuum, insulator, and emitter material intersect can also trigger these effects [221], [222]. The probability of leakage currents and dielectric surface flashovers depends largely on the length of the leakage path and the adsorbate density on the surface [88], [93], [94]. Increasing the thickness of the insulating layer is a straightforward method of enhancing the dielectric surface's resistance, resulting in reduced leakage currents. Nonetheless, the parasitic emission from the triple point would still be present. Moreover, a thicker dielectric would increase the distance between the emitter and gate in common FEA designs, resulting in a higher extraction voltage, and the maximum layer thickness is hereby limited by the respective deposition or growth method.

For these reasons, a new approach is pursued in this work. The idea is to implement passivated trenches with high aspect ratios around the emitting structures to expand the dielectric surface area while minimally affecting the lateral distance between the gate and the emitters. Wet-thermally grown silicon oxide is a viable choice of insulator for this purpose. It is a commonly used insulator material in the semiconductor industry, which can be grown in precise thicknesses ranging from nanometers to approximately one micron by oxidizing silicon at elevated temperatures in either wet or dry flowing oxygen gas. Given the right set of process parameters it can conformally cover any silicon topology. The dielectric strength of thermally grown oxides can be as high as 1 kV/ μm , but is often below this value in reality [166]. Nonetheless, if the thickness is chosen accordingly, this material can supply sufficient insulation for gate voltages up to several hundred volts. The gate electrode should be a conductive layer that is compatible with CMOS processes for cleanroom processing, does not interact harmfully with the dielectric, and is not affected by subsequent process steps. For this proof of concept, thermally evaporated aluminum was chosen due to its simplicity and lack of alternative options in the cleanroom at the time. The directionality of thermal evaporation inhibits the deposition of aluminum on the vertical sidewalls of the trenches. This minimizes the risk of short-circuits and device failure caused by residual gate material on insulating surfaces after processing. The disadvantage of this material choice is the low melting point of aluminum at 650 °C [166]. Previous research has shown that this can lead to thermal evaporation and/or outgassing during device operation, which can negatively impact the FEA's lifetime [223]. Nonetheless, this study serves as a first proof of concept and can be easily replicated using alternative gate materials in the future.

Figure 6.1 illustrates the key processing steps of the envisioned fabrication process for gated SiNW FEAs in a schematic manner. Each schematic represents a cross-section of a simplified device after photolithography and the subsequent structuring of the underlying

layer. Note that the figure and corresponding description below only detail the essential process steps directly related to nanowire, gate, and insulator deposition, growth and/or etching. For a complete process flow, including all the individual steps and corresponding parameters, please refer to Table 4 in Appendix A.

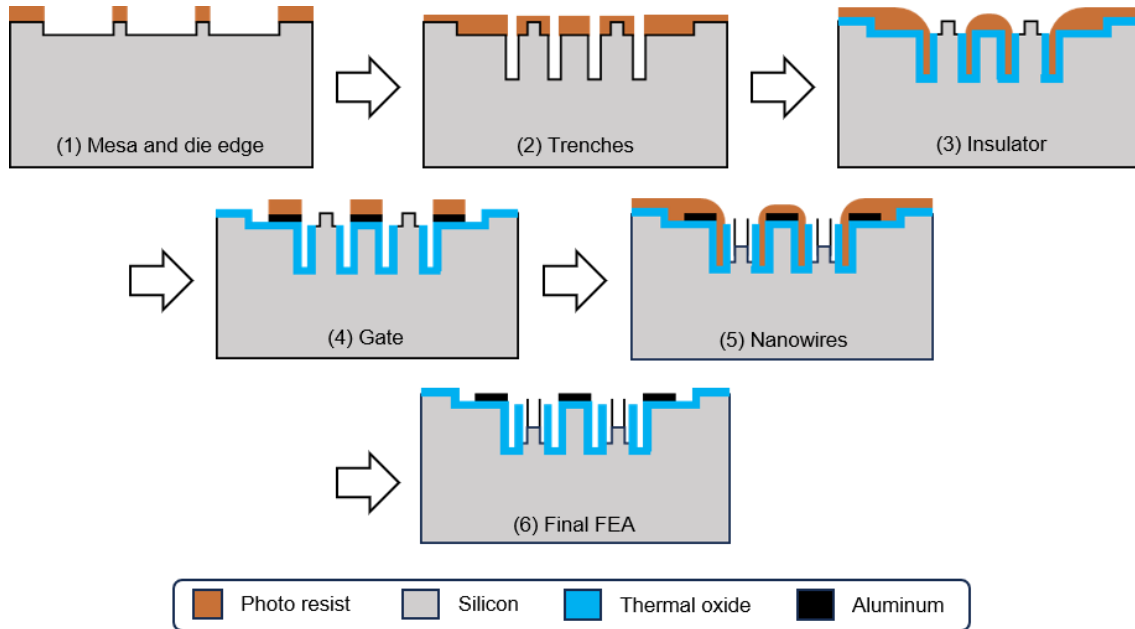


Figure 6.1 Schematic cross-section diagram of the key processing steps for the fabrication of gated silicon nanowire field emitter arrays. Each step is illustrated after photolithography and subsequent etching of the underlying layer.

Similar to the procedure for ungated SiNW FEAs, the first step is to structure mesas using the Bosch process (Figure 6.1(1)). These will later serve as initiators for the nanowire formation. The lithography mask for this process is the same as that used for the ungated SiNW FEAs. Next, a second lithography mask is applied to etch trenches surrounding the mesas, also using the Bosch process (Figure 6.1(2)). This is followed by the growth of an insulating layer, which is realized in this case by wet thermal oxidation of silicon, as mentioned before. A third lithography mask is used to wet-chemically etch the silicon oxide from the top of the mesa structures with buffered hydrofluoric acid, while maintaining the passivation of the trenches (Figure 6.1(3)). Subsequently, the gate material is deposited on top of the insulating oxide by thermal evaporation of aluminum. A fourth photolithography mask is used for wet chemical etching the aluminum with a phosphoric acid based etching mixture (Figure 6.1(4)). The final step is the nanowire formation using an unmasked Bosch process on the mesas. To protect the surrounding structures, the oxide etching mask can be reapplied (Figure 6.1(5)).

In total, the fabrication therefore requires a set of four different lithography masks for the five key process steps. Figure 6.2(a) provides an overview of the overlaid mask set that

was designed for this purpose. Note that the mask for the trenches is depicted in negative polarity, which means the turquoise areas are transparent on the mask, and the rest is opaque. All the other masks are in positive polarity. Each die on the wafer measures 7 mm x 7 mm, with a standard active area of 4 mm x 4 mm. Exceptions to this can only be found in the fourth and ninth column of the design. Here, each die contains either six different FEAs each with an active area of 1 mm x 1 mm (labeled as '6X1X1' in Figure 6.2(a)) or ten individual cells of different FEA types (labeled as 'SINGLE' in Figure 6.2(a)). As already explained in Chapter 6, the masks feature various FEA layouts, whereby the nomenclature of the types is based primarily on the mesa shape and diameter. On the left-hand side of the masks, there are line-shaped mesas with widths of either 5 μm or 10 μm , labeled 'LD5' and 'LD10,' respectively. Additionally, FEAs with a 50 μm wide mesa in which the gate electrode runs through the center are located here. The purpose of this design was to symmetrically surround a single row of nanowires with the gate aperture, rather than the usual two parallel rows. These emitter types are referred to as 'LS'. On the right-hand side of the mask, circular mesas with diameters of either 5 μm or 10 μm , denoted as 'CD5' or 'CD10', are found. In addition to mesa dimensions, the gate aperture diameter was varied to establish feasible combinations that account for the alignment accuracy limitations of the mask aligner and the respective operator. This adds another parameter to the naming convention, which is labeled with the letter 'G' followed by the distance in micrometers between the nanowires and the inner edge of the gate. There are three possible values for this parameter: 3 μm , 5 μm , or 7.5 μm . For clarity, Figure 6.2(b,c) shows a complete lateral dimensioning of two individual cells of a CD5G5 or LD5G5 emitter, respectively. Note that the mask set design has a nominal overlap of 2.5 μm between the gate and the trenches for each type, facilitating self-alignment of the masks as long as the misalignment does not exceed this value. The insulation of the trenches is not affected by this, since the typically highly directional aluminum deposition by thermal evaporation prevents coating of the vertical sidewalls. The dimensions of a single cell for FEAs with circular mesas measure 40 μm x 40 μm , resulting in a total of 100 x 100 individual cells on a standard active area. Line-type cells measure 40 μm horizontally and the full length of the active region vertically, resulting in 100 cells per die. The magnification in Figure 6.2(a) reveals, that the various types are grouped in sets of six, which are equally distributed across the wafer. This compensates for any position-induced inhomogeneities in nanowire formation resulting from the radial dependence of the RIE process, that was previously mentioned in Chapter 5.2.

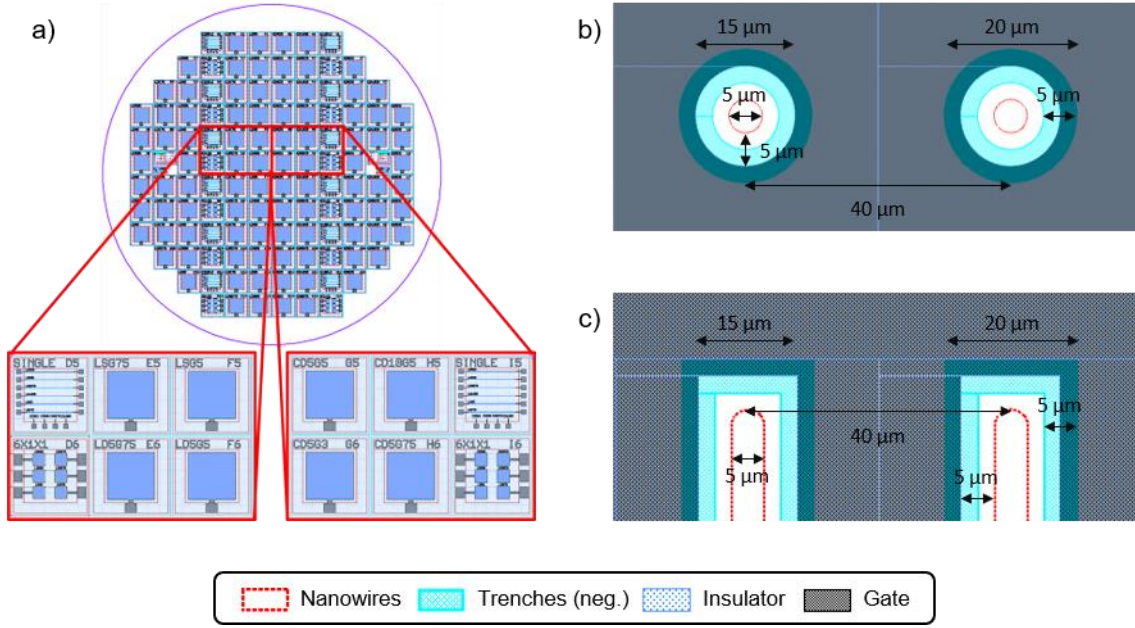


Figure 6.2 (a) Overview of the mask set design used for the gated SiNW FEA fabrication. The set consists of four different masks. The trenches mask is illustrated here in negative polarity while the others are in positive polarity. (b) and (c) show enlarged images of two single cells of a CD5G5 and LD5G5 FEA, including all relevant dimensions.

A special feature of this concept and mask layout is, that it may also be utilized to integrate a gate for TMAH FEAs. However, the following process implementation and optimization focuses on the gated SiNW FEAs, as these are the more promising candidates to fulfill the application requirements. For preliminary and unoptimized results on gated TMAH FEAs, please refer to Appendix C.

6.2 Process Implementation and Optimization

The first step in developing a working process flow for gated silicon nanowire FEAs is to determine appropriate values for dimensions that were not specified by the lithography masks. The height of the mesas, the trench depth, and the silicon oxide thickness are the essential ones to consider. The height of the mesas primarily defines the height of the edge surrounding each die. Greater height leads to more embedded structures and easier handling of samples. However, elevated die edges affect the subsequent lithography steps by introducing an unwanted gap between the mask and the exposed surfaces in contact mode exposure. To minimize the effect on exposure distance, a height of 5 μm was considered a reasonable tradeoff since it is below the 8 μm thickness of the photoresist. The trenches were etched to a depth of around 20 μm to avoid possible short-circuiting of the FEA as a result of non-ideal gate evaporation directionality, and to enable efficient suppression of leakage currents across the trench surface length. For the initial experiments, a silicon oxide thickness of 800 nm was

selected as this could be homogeneously and conformally grown on the pre-structured topography. The thickness allows the potential application of high extraction voltages, which helps to determine the maximum current limits of the fabricated emitters.

Based on this plan, the parameters for each of the 23 individual process steps (see Table 4, Appendix A) were first roughly determined and then gradually optimized through multiple test runs. The resulting structures were examined using SEM inspection. Once the desired morphology of the FEAs was achieved, the most promising samples were electrically characterized in triode configuration measurements. An example of a sample produced using this optimized process flow can be seen in Figure 6.3(a). It depicts a cross-sectional SEM image of an individual LD5G75 cell. Note that resist residue is visible in the trenches of this otherwise good sample, which is why it was selected for the destructive cross-sectional analysis.

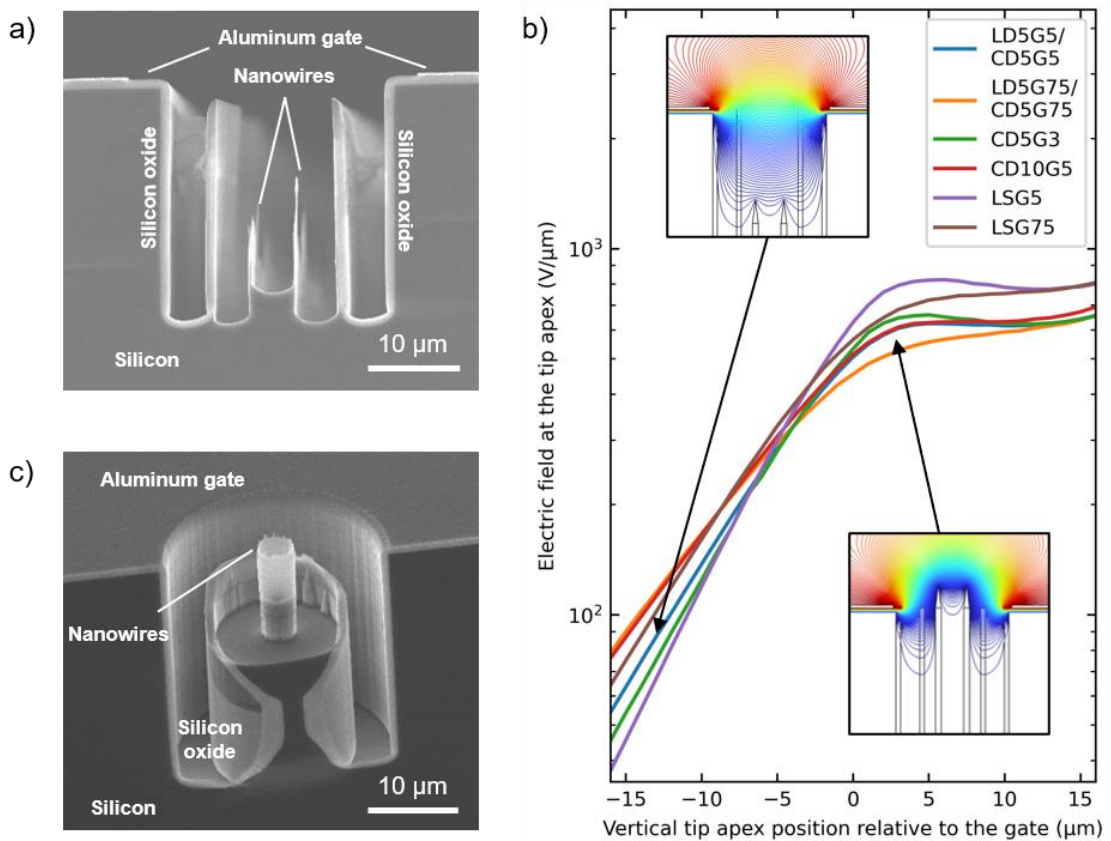


Figure 6.3 (a) Cross-sectional SEM image of a single LD5G75 cell resulting from the initially established process flow. (b) Results of an electrostatic 2D simulation illustrating the electric field strength at the nanowire apex versus the tip's vertical position relative to the gate for various FEA types. The insets demonstrate the equipotential line distribution of two selected data-points of the LD5G5 graph. (c) Cross-sectional SEM image of a CD5G75 FEA cell after the process flow was adjusted to elevate the nanowires and enhance the electric field at the apex.

The positive outcome of the preliminary electrical characterization is, that the majority of samples did not show any gate current for extraction voltages up to 500–600 V. This indicates

that the fabricated structures might effectively prevent leakage currents and dielectric breakdown of the insulator within this voltage range. This voltage range exceeds typical operating ranges of state-of-the-art devices, as detailed in Chapter 3.2, which usually range up to 100 V and occasionally up to 300 V [110], [157], [159]–[161]. However, none of the measured samples exhibited a detectable emission current at the anode either. One possible explanation for this is the vertical positioning of the nanowires in relation to the gate. As can be seen from Figure 6.3(a) the emitting structures have intentionally been positioned significantly below the surface of the die to protect the delicate nanowires during sample handling. However, this placement might result in electrostatic shielding of the emitters by the surrounding bulk silicon underneath the gate. To investigate this, a two-dimensional simulation study was performed using the electrostatic module of Comsol Multiphysics® [224]. In this study, single cells of the various FEA types were modeled according to the dimensions and materials defined above and by the lithography masks. The nanowires were approximated by cylindrical pillars with a diameter of 1 μm and variable height. On top of these pillars, there are cones with a fixed height and tip radius of curvature of 4 μm and 10 nm, respectively. This way, changing the height of the nanowires will not affect the shape of the tip, and consequently have less impact on the FEF. The bulk silicon and the nanowires were grounded, and the gate potential was set to 500 V. The upper boundary of the model was placed at a 30 μm distance from the gate with a potential of 509 V. Thus, the electric field between the gate and the boundary is approximately equivalent to that between the gate and the anode in the measurement setup, as the anode is located about 1 cm away and is typically operated at 2–3 kV. The model was then used to investigate the electric field at the apex of the nanowires as a function of its vertical position relative to the gate plane by varying the height of the nanowire pillars. The findings of this study are illustrated in Figure 6.3(b). The insets show the equipotential line distribution of an LD5G5 cell for two different nanowire positions. Note that the absolute values of the simulated electric field strengths at the tip are significantly lower than the typically minimum required field strengths for field emission of 1 V/nm (see Chapter 2.2). The reason for this is the lack of a third dimension in the model, which means that the tips are equivalent to edges in electrostatic terms. However, the qualitative course of the simulated field strength is expected to be similar in both two and three dimensions. Regardless of the FEA type, the strength of the electric field at the emitter tip steadily increases with height until it reaches a local maximum once the apex of the nanowires lies a few micrometers above the gate plane. Subsequently, further height increase reduces the gate-induced field strength until the electric field of the anode prevails. These findings correspond with those reported in the literature [7], [225]. The reason for the initial electric field increase, mostly exceeding an

order of magnitude in these simulations, is particularly evident from the insets of Figure 6.3(b). The surrounding bulk silicon's exposed edge underneath the gate is at the same potential as the nanowires, thereby hindering the gate's electric field from penetrating deep into the cell cavity. Moving the tips closer towards the gate, as it is the case for the CD5G75 sample shown as an SEM image in Figure 6.3(c), therefore reduces the effect of this electrostatic shielding.

To elevate the emitter apex position, the number of cycles for the DRIE processes of both the mesa and nanowire formation were adjusted in several trials. The success of this process adjustment was finally verified by a comprehensive SEM analysis of multiple entire wafers. The optimized process flow did not eliminate the variation in density and shape of resulting nanowires among different FEA layouts or among FEAs of the same type located in different positions on the wafer. This is still attributed to the loading effect and the two-inch ICP coil of the RIE system and would need FEA type specific process optimization. Nevertheless, the process yields promising structures for most of the FEA layouts already. Figure 6.4 shows two SEM images each for an LD5G75 and CD5G75 sample fabricated using the final process parameters, which are detailed in Table 4 in Appendix A. Additionally, a digital microscope image of the CD5G75 and the corresponding cross-section schematic is included in the figure.

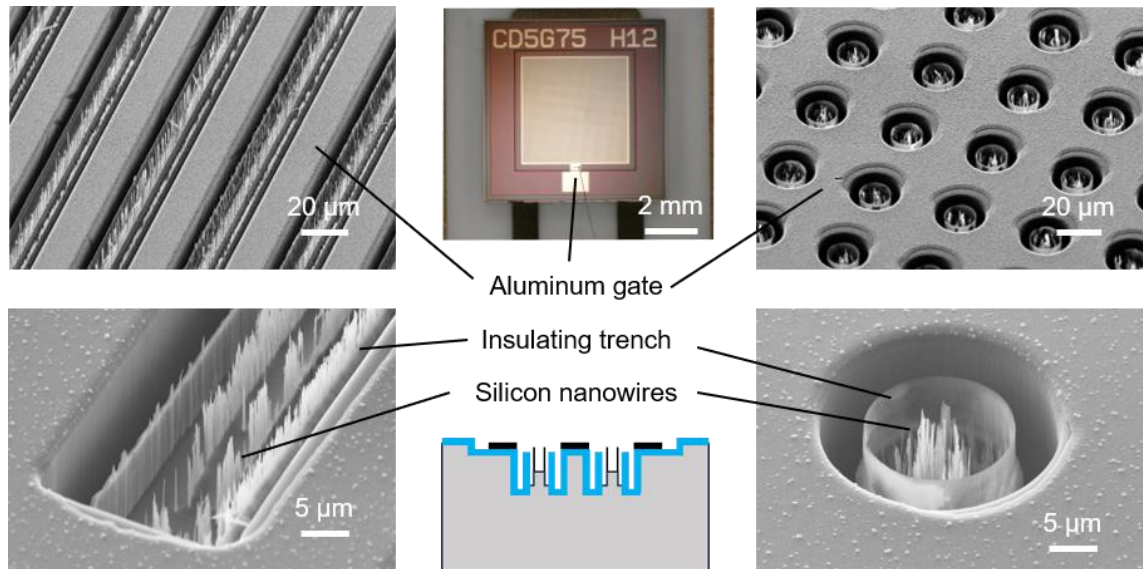


Figure 6.4 SEM images of a LD5G75 (left) and CD5G75 (right) sample fabricated with the final process flow for gated silicon nanowire FEAs. In the top center is a macroscopic image of the CD5G75 sample taken with an optical microscope. The bottom center depicts the cross-section schematic for both types.

In all cases, the nanowire tips are now much closer to the gate plane than before, which is expected to increase the extraction field strength at the apex. The high magnification SEM images of Figure 6.4 clearly show that the trenches are generally completely free of aluminum

or other residues, which should ensure sufficient electrical insulation and suppression of leakage currents. On the other hand, silicon residues can be found at the interface between silicon and silicon oxide on the side of the nanowires, particularly for line-type FEAs. However, these often also form nanowires, which could be beneficial for the emission current. The overview image of the CD5G75 sample shows what happens when the misalignment of the gate mask to the trench mask exceeds the nominal overlap. Even in this case, however, no aluminum could be detected on the sidewall of the trenches. A problem resulting from the elevated nanowire position is indicated in the overview image of the LD5G75 FEA. The reduced embedding in the cell cavity exposes the nanowires to mechanical damage, causing them to bend and break more frequently. In the worst case, a single loose nanowire can cause a short circuit between the silicon and the gate, resulting in failure of the entire FEA. Conductive dirt particles, which are difficult to avoid when handling samples outside a cleanroom environment, can also lead to this result just by covering one single cell of the FEA. For these reasons, careful and clean sample handling must be ensured during wafer dicing, SEM inspection and electrical characterization.

6.3 Electrical Characterization

For the electrical characterization of the gated SiNW FEAs, low n-doped (phosphorus) <100> silicon wafers with a resistivity of 10–20 Ωcm were processed following to the process flow outlined in Table 4 of Appendix A. After a subsequent SEM analysis, the samples underwent a short circuit test using a semi-automatic wafer prober before being transported to the measurement setup. During the initial test measurements in the setup, a significant number of the measured emitters were found to have a short circuit directly at the beginning or shortly after the start of the electrical characterization. This occurred even though no irregularities were identified during the SEM analysis and preliminary short circuit testing. Handling errors or possible electrostatic discharge damage during transport between laboratories may explain this phenomenon. Furthermore, the choice of gate material also appears to have an influence on this, as will be discussed in more detail later in this chapter. In any case, due to this low yield, only individual measurements of two CD5G75-type samples are discussed here to demonstrate the functionality and potential of the developed FEAs. A comprehensive analysis of the various structures on a statistical basis will be part of future work once the yield is optimized. All measurements were carried out in triode configuration at a base pressure of 10^{-5} mbar with a constant anode potential of 2 kV. To assess the effectiveness of the trenches under harsh conditions, no pretreatment was applied to the samples to remove the surface adsorbates resulting from regular air storage.

The first factor to be investigated is the transmission rate of electrons through the gate, as it impacts the electron source's power consumption, controllability, and lifetime. It can be determined by the current component arriving at the anode and/or the current component intercepted by the gate electrode. Based on the preceding section's preliminary measurements, it is advisable to keep the operating range of the gated SiNW FEAs below 500 V to effectively suppress leakage current and prevent dielectric breakdown of the insulator. Therefore, to avoid current measurements on high floating potentials, the high voltage source of the extractor (HV_1 of Figure 4.5) can be replaced with a Keithley 6487 picoammeter/voltage source [226], which is capable of output voltages up to 500 V. However, this configuration carries the risk of damaging the measuring instrument due to arcing from the high-voltage anode to the gate potential. To mitigate ionization of the residual gas and local pressure increase by potential outgassing and thermal evaporation of the aluminum anode by the electron beam, which is a known issue in the literature [162], a maximum emission current of 10 μA is imposed in the following transmission measurement. Apart from this, the transmission evaluation follows the CDLT methodology with the three-part sub-measurements, consisting of IV characteristics before and after a one-hour constant current measurement at different set current levels. After the measurement procedure the sample was exposed to air for several days before it underwent a voltage sweep in reverse polarity to investigate the potential impact of leakage currents resulting from adsorbates on the insulator surface. No current flow from gate to emitter could be measured within the voltage range of the transmission measurement. This indicates effective suppression of these currents by the trench under the given measurement conditions.

The primary focus of this transmission study is to analyze the constant current transmission, as this is fundamental for future applications. To examine the phenomena occurring during this type of operation in detail, Figure 6.5(a) displays the CCM results of three out of the nine sub-measurements from the measurement. The current flow into the FEA (I_{emi}) is shown in red, while the measured gate current (I_{gate}) is depicted in orange. The transmission rate (T), which is illustrated in blue, is hereby calculated using the formula $T = 1 - I_{\text{gate}}/I_{\text{emi}}$. Additionally, the extraction voltage applied to the FEA is plotted in gray. Figure 6.5(b) presents a summary of all sub-measurements by plotting the mean transmission rate for each current level and displaying the corresponding standard deviation as error bars. Additionally, the mean extraction voltage for each one-hour CCM is shown, to demonstrate the operating range of the FEA. Other than in the diode configuration measurements, the electric field is not specified in this case, since the geometry is more complex and the apex field may also be determined by the anode potential.

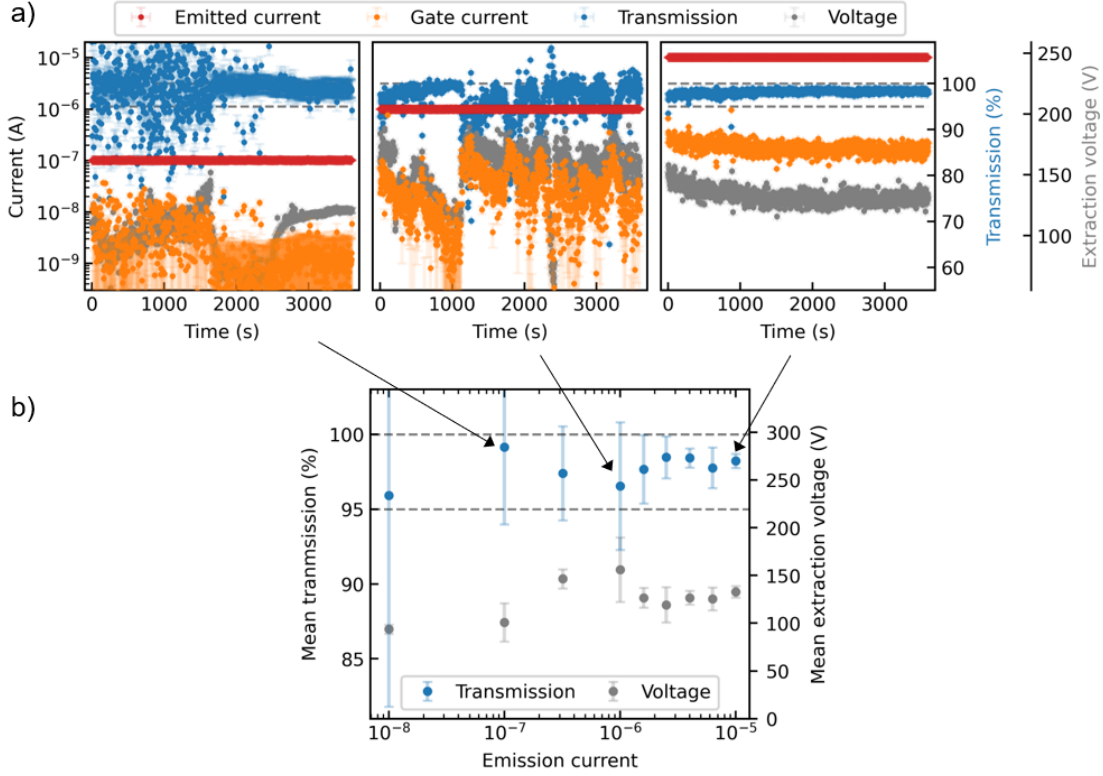


Figure 6.5: Transmission measurement results for a CD5G75-type gated SiNW FEA sample. (a) illustrates the current flow from ground potential to the FEA (red), along with the current component intercepted by the gate (orange), the corresponding transmission rate (blue), and the extraction voltage (gray) for three individual one-hour constant current measurements at set current levels of 100 nA, 1 μ A and 10 μ A. (b) presents the mean transmission rate (blue) and required extraction voltage (gray) for each sub-measurement as a function of the corresponding set current level. The gray dotted lines in both graphs indicate the desired transmission rate target range of 95–100%.

The average transmission rate consistently exceeds 95% for each sub-measurement, regardless of the current level. The mean extraction voltage tends to remain steady between 100 V and 150 V, despite a minor increase with current. Notably, the standard deviation of the mean transmission decreases as the current increases. This decrease can be partly attributed to the gate current measurement's constant noise level, which is in the low nanoampere range. It also explains the physically unreasonable transmission rates exceeding 100%, which can be seen in Figure 6.5(a), as these result from noise induced negative gate currents. With increasing emission current, also the gate current increases, until the effect of the constant measurement noise level vanishes. Another possible factor impacting the standard deviation of the mean transmission is the activation, conditioning, and failure of individual emitters or entire cells of the FEA. These effects are particularly noticeable in the 1 μ A CCM depicted in the middle part of Figure 6.5(a). Note, that the extraction voltage follows the course of the gate current almost congruently in these triode configuration measurements most likely due to the interaction of the anode and extractor fields. In the beginning of the

1 μA measurement, the extraction voltage steadily decreases for roughly 1100 seconds, which can be attributed to conditioning effects of the active emitters, as detailed in Chapter 4.2.3 or in [83], [187], [188]. This decrease in voltage also reduces the lateral component of the electric field at the emitter apex, which results in a decreasing gate current and a corresponding increase in transmission. After about 1100 seconds of the measurement, there is a sudden rise in gate current and voltage, combined with a significant transmission rate decrease. One possible explanation for this is that only a few individual cells of the FEA carry the entire emission current at this moment. If one of them is defective, the gate voltage must rise to activate new emitters and/or entire cells to compensate for the loss, thereby consequently increasing the gate current. The newly activated emitters are then conditioned again, triggering a decline in voltage and an increase in transmission. This process is repeated multiple times, especially during the initial sub-measurements at low and medium currents. As the FEA operation time progresses, typically the total number of cells emitting in parallel gradually increases, leading to a more even distribution of the current load. This stabilizes the transmission rate, as evidenced in the 10 μA measurement in Figure 6.5(a) and by the decreasing standard deviation with increasing emission current in Figure 6.5(b).

To investigate the potential failure of individual FEA cells, SEM examination of the CD5G75 sample was performed following electrical measurement. Figure 6.6(a) provides an SEM image overviewing the sample's active area, while Figure 6.6(b) and (c) present magnified images of multiple or individual cells. A significant number of cells display signs of degradation after the measurement, predominantly in the shape of melting traces on the aluminum gate. In some cases, the silicon structure that supports the nanowires has vanished entirely. The extent and appearance of these degradation traces suggest that the main cause of the destruction is arcing between the emitters and the gate. This is supported by the fact that the low melting point of the aluminum gate increases the risk of localized pressure buildup due to gate material evaporation, as previously mentioned in Chapter 6.1. Furthermore, the images illustrate that the liquefied aluminum is spread over large areas, which can be attributed to the power dissipation by the flashover. Despite the considerable number of cells and gates that literally exploded, this emitter did not experience a short-circuit during the measurement. This suggests, that the insulating trench surrounding the emitters can reduce the risk of conductive interconnections between the gate and the silicon substrate. However, as mentioned at the beginning, a significant number of tested emitters short-circuited after a short measurement time during the initial test measurements of the gated FEAs. Accordingly, the lifetime of the individual sample seems to depend on chance at this point.

Therefore, future work should consider alternative choices of the gate material, as this could significantly increase both the FEA's lifetime and the yield of the fabrication process.

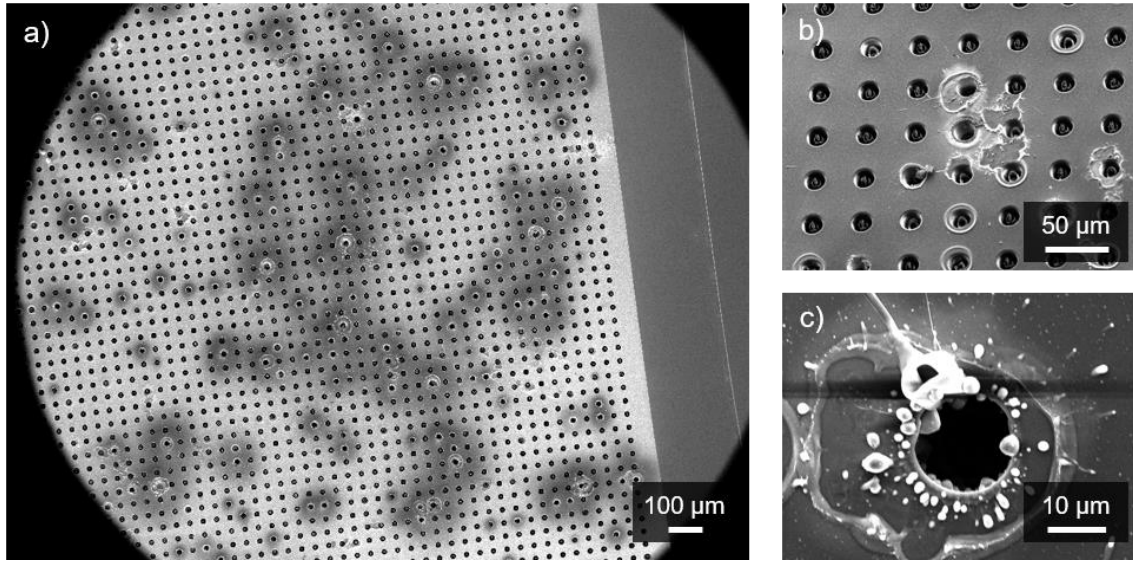


Figure 6.6: SEM images of the gated SiNW sample after the transmission evaluation measurement.

The next step is to investigate the maximum emission current capability of the gated SiNW FEAs. For this, the setup is built back to its original configuration, as described in Chapter 4.2.1, and a new CD5G75 sample is installed. Without the picoammeter, only the current monitor of the anode power source is available to evaluate the transmission rate of electrons through the gate. Even though it has a limited resolution and accuracy, it can be used to identify short circuits or major leakage currents occurring during the measurement. Figure 6.7 illustrates the measurement results obtained by the CDLT for the second CD5G75 sample, measured at a chamber base pressure of 10^{-5} mbar. Note that the measurement was manually aborted after the 630 μA sub-measurement due to a malfunction in the gate voltage supply. Even though it was set to a constant value of 500 V, it started increasing and fluctuating significantly during the 400 μA und 630 μA CCM. This created a risk of damaging the control circuit due to excessive voltages. Additionally, determination of the effective extraction voltage applied to the emitter was affected by the fluctuations, as can be seen for the measurements in question in Figure 6.7(b). The most likely cause of this malfunction is a flashover from the anode to the gate potential, possibly triggered by outgassing of the anode. Nevertheless, the control circuit was still able to consistently maintain the designated emission current until the end of the entire CDLT.

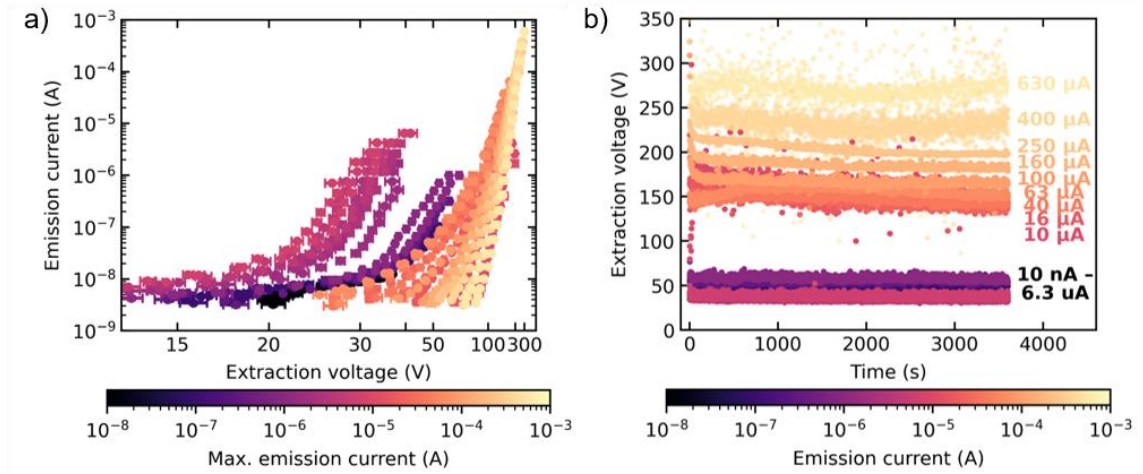


Figure 6.7: CDLT results for a CD5G75 gated SiNW FEA sample. (a) illustrates the IV characteristics before and after each constant current measurement in ML coordinates. (b) shows the corresponding course of the extraction voltage during each CCM.

Noticeable in Figure 6.7(a) is the non-linearity of the IV characteristics with maximum emission currents up to 10 μ A. As mentioned in Chapter 2.4, this is typically an indicator for the presence of leakage currents into the gate. The extraction voltages during the respective CCMs shown in Figure 6.7(b) are remarkably low compared to the previous measurement, confirming the suspicion, that these measurements exhibit a combination of leakage and field emitted current. Flashover induced aluminum splashes, as shown above, could be a possible cause of such leakage currents. However, since the leakage currents are present from the beginning, it is more likely that they were caused by contamination of the sample during installation in the measurement setup. During the characteristic curve before the 10 μ A CCM, the first measurable currents can be seen on the anode current monitor and the non-linearity of the curve decreases for this and the subsequent measurements. Furthermore, the extraction voltages increase significantly during the sweeps and at the beginning of the CCM at this current level. The combination of the shape of the characteristics, the increase in voltage, and the evidence of transmitted current suggests the presence of both leakage and emission current for these current levels. The proportion of leakage current in the total current can vary greatly, as leakage paths may degrade and disappear during measurement due to excessive current densities. Conversely, flashovers, charging of the insulator surface and other degradation effects can create new leakage paths of varying extent. Furthermore, according to literature, upward bending of FEA characteristic curves can also be caused by the superposition of many individual emitters with different field enhancement factors, independent of leakage currents [44]. Unfortunately, the anode current measurement is very noisy here, which is why

the absolute value of the transmission cannot yet be used as a reliable validation here. Therefore, a more precise measurement of the transmitted current is required to draw clear conclusions in the current range between 10 and 100 μA .

For set current levels of 100 μA and above, however, the IV characteristics in Figure 6.7(a) exhibit a linear trend, indicating negligible impact of potential leakage currents into the gate. The extraction voltages during the corresponding CCMs range reasonably from 150–300 V, despite the power supply fluctuations during the last two set current levels. The slope of the extraction voltage curve during the CCM is negative up to and including the 250 μA measurement. The last two current levels should be interpreted with caution due to the fluctuations in the gate voltage supply. However, a slightly negatively sloped to horizontal curve is indicated here on average as well. Additionally, there is a barely perceptible offset between the characteristic curves before and after the CCM. This suggests a very low degradation of the gated SiNW FEA, even at high currents and a pressure level of 10^{-5} mbar. The transmission rate, as determined by the anode current monitor, is consistently slightly above 100% in this current range. One possible explanation for this phenomenon is the ionization of the residual gas, possibly enhanced by outgassing of the anode due to the emission current. Due to the high anode potential and the associated electron energies, it is conceivable that a single electron causes several ions, for example through impact ionization, which subsequently increases the number of charge carriers arriving at the anode. However, it is questionable if the resulting ion currents could notably impact the emission current at this magnitude [227]. Alternatively, the limited accuracy and resolution of the anode current measurement may also contribute to this phenomenon. Nevertheless, based on the measured anode current and the linear characteristics, the electron transmission rate can be presumed to be reasonably high. Therefore, the measurement demonstrates that these emitters can operate for at least five hours within the emission current range of 100–630 μA at a pressure of 10^{-5} mbar without experiencing device failure, despite the premature termination of the measurement due to a malfunction of the setup.

6.4 Conclusion and Outlook

In summary, the gated SiNW FEAs consistently demonstrated an electron transmission rate of over 95% for emission currents up to 10 μA , regardless of the current level. Fluctuations observed at lower set current levels can be attributed to the failure of active emitters or entire cells of the FEA. However, these fluctuations stabilize over time probably due to the gradual activation of new cells during the measurement. The choice of gate material seems to be a major factor contributing to the degradation of gated FEAs, due to the low melting

point of aluminum, which increases the probability of flashovers caused by localized pressure buildup through outgassing. Nevertheless, the maximum current measurement based on the CDLT methodology revealed promising initial results for this proof of concept. The sample under investigation was operated for over 24 hours at a base pressure of 10^{-5} mbar and various current levels. A maximum emission current of 630 μA was achieved and attained for one hour, before the measurement had to be manually terminated due to a setup malfunction. The corresponding current density of 3.9 mA/cm^2 , could be maintained for the entire hour at a mostly constant voltage level of about 280 V, despite the fluctuating voltage source. During the other constant current measurements, no significant increase of the necessary extraction voltage could be detected either, which indicates an excellent current carrying capacity and low degradation probability of this emitter type. The obtained maximum current density for constant current measurements is still below the state of the art in UHV conditions. However, as comprehensively discussed in Chapter 3, comparing field emission measurements under different measurement environments, especially with different operating pressures and modes, is unreasonable. Furthermore, the measured sample was still functional even after the last five hours of operation at constant emission currents above 100 μA . This makes them a good basis for application tests in field emission-based X-ray tubes at this stage of development. In addition, there is still a lot of room for optimization to achieve the lifetimes required for commercial applications.

Firstly, in future devices, the gate material should be replaced with materials that have a higher melting point. Polycrystalline silicon is a common choice due to its high process versatility, high melting point, and good conductivity when doped [133], [161], [166], [228]. Secondly, a conditioning procedure could be performed before device operation to support cell activation and potentially improve the FEA's lifetime. Possible pretreatments for clearing the surfaces of adsorbates include exposure to UV light [94] or heat [90], [91], [93]. Another option is electrical conditioning, achieved through a careful first switch-on procedure [21]. In the long term, the emitter density, which was conservatively chosen here, can be increased. For comparison, the pitch between the individual cells of *Guerrera's* silicon FEAs, which were presented in Chapter 3, is 1 μm [161]. In contrast, the FEAs presented here have a lateral distance of 40 μm between the individual gate apertures. Moreover, to prevent device failure due to short-circuiting by contamination during sample handling, an insulating layer can be introduced on the sample surface, covering the gate. This would also further reduce the risk of short circuits during the measurement. Furthermore, additional electrode layers could be implemented on top of this insulator. These can then be used, e.g., to focus the emitted electron beam [106], [212].

In addition to optimizing the device, the measurement setup must also be optimized for transmission measurements at high currents. One potential improvement would be to introduce a reliable method for measuring anode currents, such as measuring the voltage drop across a shunt resistor. Additionally, the voltage supply of the gate should be protected against flashovers, for example, by using a quenching resistor in the measurement path. To minimize the risk of excessive thermal evaporation, ablation, and/or outgassing of the anode at high current densities, it is advisable to substitute the aluminum anode with a more robust material. Furthermore, the implementation of a cooling system could be advantageous in compensating for the dissipated power.

The optimized setup can then be used to perform further characterization measurements of the gated SiNW FEAs. More specific investigation is needed to determine the effectiveness of the trenches in suppressing leakage currents. Moreover, it is necessary to identify the most promising emitter design among the different types based on statistical measurements. Once this is found, the fabrication process can be further optimized with regard to the nanowire density and length. Additionally, as suggested by the results from Chapter 5.3.2, the influence of different doping types and densities, in combination with the morphological current limitation of the nanowires, may have the potential to further optimize the lifetime of the FEAs. To evaluate saturation effects and their impact on current distribution in the array, it would be advantageous to quantitatively measure the emission current of individual emitters during integral operation of the FEA. The following chapter discusses the development of a novel measurement technique to achieve this goal.

7 Quantitative Field Emission Imaging: A Novel Method for Characterization

As mentioned before, there are numerous reasons that can cause the defect of an FEA, including melting or thermal explosion of emission sites [3], [116], [200], [201], [203], [204], [207]–[209], changes in the tip geometry due to surface migration [229] and/or ion bombardment [202], [203], or contamination of the tips surface with adsorbates [3], [83], [204], [205]. Many of these failure mechanisms can be traced back to non-uniform current distribution, caused by geometric inhomogeneities of the tips in the array [145], [161], [213]. There are approaches in the literature to improve the emission distribution, e.g. by process optimization [213], [230] and morphological [145], [161], [210], [231] and/or doping-dependent [75], [149], [155] current limitations. However, for a robust and detailed analysis of the effectiveness of these methods, it would be necessary to measure the emission current of each individual emission site of the array in-situ and in a spatially resolved manner during the integral operation of the FEA. As described in the following section, there is currently no existing measurement method that fulfills all of these requirements simultaneously. Therefore, this work presents a new measurement method, which is comprehensively explained in this chapter's scope, starting with an overview of the current state-of-the-art metrology.

Note, that the basic methodology of the herein presented measurement method and some of the results discussed in this chapter have already been published in [102], [103], [232] and are marked accordingly.

7.1 State-of-the-Art Metrology

A relatively simple and frequently chosen way to measure emission uniformity of an FEA is provided by observing the cathodoluminescence of phosphor screens [7], [201], [233]–[239]. For instance, *Lui et al.* [233] used them to characterize the turn-on field distribution of CNT cathodes by simply counting the simultaneously appearing luminous dots on the screen. However, this provides only a qualitative overview rather than a fully quantified emission current distribution. A more sophisticated approach is being pursued at the Ioffe Institute in St. Petersburg, Russia [201], [234], [240]–[243]. Based on a USB microscope, they observe the luminescence patterns of a glass anode coated with ITO and luminescent powder [241], [243]. By computerized numerical evaluation of these patterns they analyze

the current distribution and emission uniformity of multi-tip field emitters [201], [234], [235], [241]. However, the limited linearity and dynamic range of the cathodoluminescent screens can be a problem here, especially for emission currents that can vary over several orders of magnitude [244]–[247]. In addition, high currents typically lead to short lifetimes of the expensive screens and the maximum spatial resolution depends strongly on the coating properties and grain size [7], [236], [248]. Especially in doping-dependent studies, the luminescence of the screen can also influence, for example, saturation properties of p type silicon FEAs [70], [74], [75].

An alternative and also widely practiced approach is to measure each emission site in the FEA on its own, using a field emission scanning microscope [7], [249]–[251] or scanning anode field emission microscope [97], [248], [252], [253]. *Rughoobur et al.* [97] used this method recently to investigate the integrated resistive current limiting in silicon field emitters on nanopillars. The paper shows that measurements of this type allow very detailed analysis of the emission from each emitter in the FEA with lateral resolutions in the range of a few micrometers. However, very close attention must be paid to various parameters such as the distance from anode to tip and the radius of curvature of the analysis tip in order not to falsify the measurement results. Apart from that, this measurement method requires very expensive and highly complex equipment, as well as a lot of measuring time. However, the biggest disadvantage is that the measurement does not show the behavior of the individual tips in integral operation. This can deviate considerably from the individual emission under ideal conditions, for example due to thermal or electrostatic interaction of neighboring emitters in close proximity [54], [98]. Apart from this, one can find sporadic creative approaches in the literature. For example, *Hobbs et al.* [254] used electron-beam resist for superimposed analysis of all emission processes in FEAs. However, the exposed result does not give any information about the temporal behavior of the emission distribution, but only reflects an integrated image.

The quantitative field emission imaging (QFEI) measurement method presented in this and subsequent chapters has the potential to combine the advantages of all the above methods in a simple way that can be implemented inexpensively in any basic vacuum measurement setup. Its simplest implementation requires just a commercially available Raspberry Pi HQ camera [178], which is becoming increasingly popular in the scientific community due to its well-documented quasi open source programming and low price [255]–[257]. However, alternative detectors can be employed and the setup can be enhanced in various ways depending on the specific application. The results presented herein serve as proof of concept and the initial development phase of this innovative measurement approach. The technique allows

for highly resolved mappings of emission currents across multiple orders of magnitude, thanks to the sensor's high resolution and the ability to expand the dynamic range through varying exposure times. By employing python-based evaluation algorithms, the method permits quantitative reconstruction of individual tip emission currents. This can be utilized to statistically analyze the emission distribution in the array. The source code developed as part of this thesis for controlling and coordinating the camera and measurement setup, as well as for analyzing the data, are planned to be published publicly. Together with the documentation in this chapter, an easy reproduction of the measurement setup for others should be guaranteed to allow for an establishment of the measurement method as a potential standard in the field emission community.

7.2 Basic Methodology

This chapter is based on my journal article published in *J. Vac. Sci. Technol. B*, vol. 40, no. 1, p. 014202, Jan. 2022, doi: 10.1116/6.0001551. It also includes data published in my conference paper [102] © 2023 IEEE and presented at the *IEEE 36th International Vacuum Nanoelectronics Conference (IVNC)*, Jul. 2023.

7.2.1 CMOS Imaging Sensor for Field-Emitted Electron Imaging

The core part of the QFEI is the Raspberry Pi HQ camera [178], which is equipped with a back-illuminated Sony IMX477 [258] CMOS imaging sensor mounted on a PCB board. It consists of 4056 x 3040 pixels with a diameter of 1.55 μm and a sensor image area of 6.287 mm x 4.712 mm. The lateral resolution is thus high enough to compete even with the very sophisticated field emission microscope measurements [97]. In order to minimize absorptions of the signal, the sensors surface must be as exposed as possible. Therefore, the CS mount and sealing lip of the camera is removed, revealing a glass plate and spacer frame glued around the CMOS sensor. The adhesion can be reduced by heating the camera with a hot air gun, which allows the glass plate to be removed mechanically with a scalpel. This exposes the bonding wires of the sensor. To protect them from damage or electrical short circuits during assembly, they are embedded with UHV compatible and electrically non-conductive adhesive.

To clarify the question in which form electrons can be detected by the sensor, Figure 7.1 shows a cross-section analysis of the Sony IMX477, obtained by focused ion beam milling and SEM imaging, after macroscopic exposure of the sensor surface.

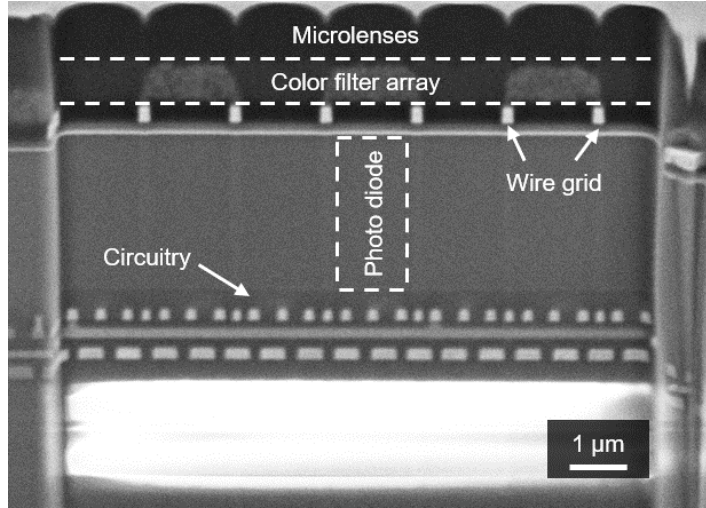


Figure 7.1 Cross-sectional SEM image of the Sony IMX477 after focused ion beam analysis.

This back-illuminated CMOS imaging sensor uses pinned photodiodes to detect the incoming signals [258]. On top of these diodes there are still multiple passive layers, such as anti-reflection coatings, color filter arrays, and microlenses [259], [260]. Note that the non-conductive materials of these elements cause a slight distortion of the vertical dimensions in Figure 7.1, due to charging effects caused by the SEM's electron beam. However, by accelerating the scan speed during image capture, this distortion can be minimized at the expense of image quality. This was used to determine the thickness of the non-conductive layers on top of the photodiodes to 1.5 μm using images with increased scan speed, which are not shown here. To evaluate whether electrons can pass through this, the Kanaya-Okayama model [261], [262] can be used. It offers the possibility to calculate the penetration depth of electrons into materials in nanometers, by using the formula

$$R(\text{nm}) = \frac{27.6AE_0^{1.67}}{Z^{0.89}\rho}. \quad (7.1)$$

Here, ρ corresponds to the material density, Z to the atomic number, A to the atomic weight, and E_0 to the initial electron energy in keV. The exact composition of the different layers in this case is not known. However, it can be assumed that they mainly consist of carbon-based polymers, as they are often resist based [263], [264]. This is also supported by the results of an energy-dispersive X-ray spectroscopy measurement, which showed that these materials are primarily of carbonic nature. Using the properties of hexagonal carbon ($\rho = 2.25 \text{ g/cm}^3$, $A = 6$, $Z = 12 \text{ g/mol}$) and an electron energy of 1 keV, the formula returns 21 nm as the maximum penetration depth of the electrons into the passive layers. To confirm this result, an additional Monte Carlo simulation of the energy dependent penetration depth

of electrons was performed using CASINO, assuming PMMA ($C_5O_2H_8$, density: 1.18 g/cm^3) as the material to be penetrated. The results are shown in Figure 7.2.

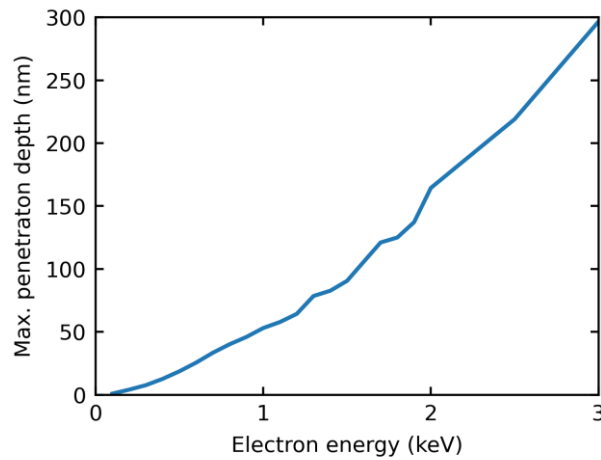


Figure 7.2 Maximum penetration depth of electrons into PMMA obtained by Monte Carlo simulation using CASINO.

This predicts a maximum penetration depth of approx. 50 nm at electron energies of 1 keV. In comparison to the $1.5 \mu\text{m}$ of passive material, it can thus be assumed with sufficient certainty that no electrons can reach the active layer of the photodiodes within the scope of the available electron energy. A more probable cause of the detector signal is bremsstrahlung, which is generated by the deceleration of the electrons when impacting the sensor's surface. The transmission rates at this X-ray energy are in the range of 50% for carbon and 60% for PMMA at an thickness of $1.5 \mu\text{m}$ (calculations based on online databases [265]–[267]). The absorption in the photoactive region of the silicon substrate, which can be estimated to $3 \mu\text{m}$ based on Figure 7.1, is above 65%. Therefore, it is likely for X-rays to be the source of the signal. Apart from this, photoluminescence of the passive layers could also contribute to signal generation.

7.2.2 Image Data Acquisition and Camera Control Software

Regardless of the origin of the signal, the optical measurement must meet two crucial conditions in order to fulfill the requirements for field emission imaging:

- 1) Linear and reproducible sensor response. Field emission currents can range over several orders of magnitude, while the dynamic range of image data is limited by the full well capacity of the pixels. By capturing multiple images at different shutter speeds this limitation can be bypassed (see Chapter 7.2.4). However, in order to bring the data to a common level, the sensor signal must be linearly related to the

integration time of the shutter and the capture parameters must be kept constant between every frame.

- 2) Fast image acquisition. Due to the fluctuating nature of field emission and the non-negligible acquisition time of the image data, multiple images must be captured per shutter speed in order to be able to counteract the fluctuations by statistical evaluation. The faster the image acquisition, the smaller the possible errors.

Therefore, several points must be considered on the software side when setting up the camera control. Regarding the reproducibility of the image data, the right choice of the camera driver is crucial to have the deepest possible access to all camera internal processes. By default, most cameras use a set of automatic parameter optimization algorithms that run on the graphic processing unit and ensure user-friendly image capturing. Access to this proprietary software is usually severely limited in order to protect the intellectual property of the manufacturers. By using `picamera2` as the camera driver, this limited access can be bypassed [268], [269]. It builds on the `libcamera` framework, which is a Linux-based and open source-friendly camera stack developed in collaboration with the industry [270], [271]. It allows the application of a tuning file, which was specifically created for scientific applications with high dynamic range [272]. It deactivates all automatic image optimization algorithms running in the graphic processing unit of the camera. Together with the self-written camera wrapper, which additionally deactivates the automatic white balance and the automatic exposure control, as well as sets the noise reduction and the analog and digital gain to one, a maximum reproducibility is ensured.

Next, an image data type must be selected. The fastest and easiest way for capturing images with the Raspberry Pi HQ camera is using the standard 8-bit JPEG file interchange format, which works efficiently due to the optimized hardware and greatly reduces the amount of data due to the compression algorithm. However, compression compromises reproducibility due to data loss. In addition, the de-Bayering and other image post-processing algorithms, such as gamma correction, are applied when requesting the standard JPEG image output, which leads to a loss of the natural linearity between exposure time and pixel charge of the CMOS imaging sensor [255].

The most reliable way to exclude any post-processing of the image data by internal algorithms of the camera is to capture the raw bitstream before it passes through the image signal processor [268]. This 16-bit I/O stream contains the pristine sensor data received by the 12-bit analog-to-digital converters [258]. However, the data is not yet sorted in a readable manner and includes a lot of information which is not needed any further. Therefore, the

bitstream must first be properly unpacked. A good description of this process can be found in [273], for example. Following these instructions returns a raw grayscale image consisting of a 4056 x 3040 array of 12-bit digital number (DN) values saved as 16-bit unsigned integers.

In order to investigate the linearity of this data type, the camera was tested in a light-proof box with a built-in light bulb as constant light source. Using the settings described above to disable all accessible image optimization algorithms, a series of grayscale images was captured in JPEG and raw format respectively. The camera's shutter speed was varied from 1 to 300 ms. In addition to the exposed series, the same measurement was also performed with the light source turned off. The three dark images for every data point were averaged and then subtracted from the respective averaged exposed images. Finally, the mean value of each array was determined to obtain the average DN of a pixel for the respective shutter speed. The error of each measurement point was determined by the empirical standard deviation and error propagation. Figure 7.3 shows the result of this measurement for 12-bit raw data obtained from the bitstream and for 8-bit JPEG data captured by using the standard procedure.

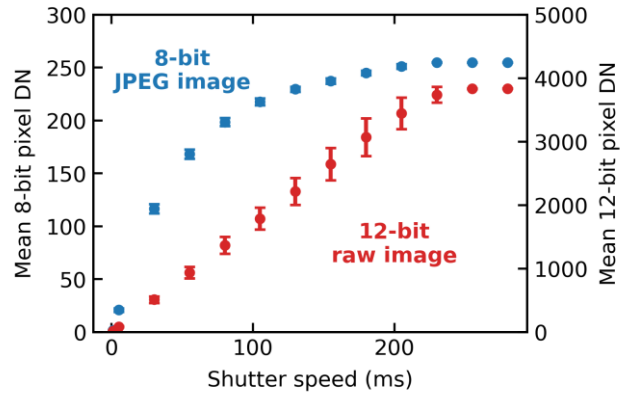


Figure 7.3 Average pixel gray value (dynamic number) for a constant light source measurement at different shutter speeds of the camera for 8-bit JPEG and 12-bit raw images.

The raw data (shown in red) show a perfectly linear sensor response up to a shutter speed of 230 ms, where it bends due to pixel saturation. Its 12-bit nature allows a maximum value of 4095 DN for a fully charged pixel. By subtracting the dark images, this value is reduced by the sensor's black level, which in this case is 255 DN. On the other hand, the JPEG measurements (shown in blue) show significant non-linearity from the outset, due to the image processing described above. Their maximum value is limited to 254 DN, which corresponds to the 8-bit maximum. The dark level has already been corrected in the post-processing, so the 8 bits are fully utilized here. Nevertheless, the 12-bit raw data allows a higher data resolution than the 8-bit JPEGs.

In terms of speed of measurement, the acquisition time is slightly shorter for raw images, although in both cases this is in the negligible millisecond range. However, JPEGs are typically between 100 kB and a few MB, depending on the information density, while the 12-bit raw data results in a fixed size of 24 MB per image. Saving such a large image to the Raspberry Pi's memory takes several seconds, compared to a few hundred milliseconds for JPEGs, making it unsuitable for the application. There are several steps that can be taken to reduce the size of the data. Given the initially large image area, it can first be cropped according to the requirements of the measured FEA. In this case, it is cropped by 50%, leaving an active sensor area of 3.1 mm x 2.4 mm and reducing the data by 75%. In addition, the resolution can be artificially reduced by summing up the grey values of 4 x 4 pixels. The addition combines the data from the 16 original 12-bit pixels into one 16-bit pixel without any loss of information besides a lower resolution. It is important to note, however, that this can cause the saturation of a single pixel from the 4 x 4 array to go unnoticed, resulting in data corruption. Therefore, during data processing, the entire 16-bit pixel is raised to its maximum value of 65535 DN as soon as even one of the pixels has reached its full well capacity. This value can then be identified in post-processing and treated accordingly (see Chapter 7.2.4). The resulting effective pixel diameter of 6.2 μm ensures a high enough resolution for the FEAs presented here (see Chapter 5.1), while reducing the data size of a single image by 94%. To preserve the life of the SD card from the many write cycles, the resulting 377 kB per image is then stored in the Raspberry Pi's RAM. By processing the image data as the shutter speed changes, the time to capture three images per shutter speed can be reduced to a total of 1.1 s. This is in the same range as the time required for JPEGs, while giving a linear sensor response in this case. To prevent the RAM from overflowing at the 1 MB/s data rate, the images are transferred to the measurement PC every 10 seconds using a Linux service asynchronously executed by the Raspberry Pi's CPU. This allows efficient data transfer without the need for additional data processing time.

Using this accelerated data acquisition method, the linearity of the sensor's response to emitted electrons must be investigated conclusively. For this purpose, the camera was mounted in a scanning electron microscope, where defined electron beams with minimum spot sizes and different currents were accelerated onto the detector at an energy of 1 keV. Three exposed and three dark images each were captured in raw format for different shutter speeds ranging from 1 to 100 ms. Figure 7.4(a) shows the resulting brightness of the emission spot after averaging and dark image subtraction of the optical data. Data points from shutter speeds where saturation of at least one pixel has occurred have been removed here.

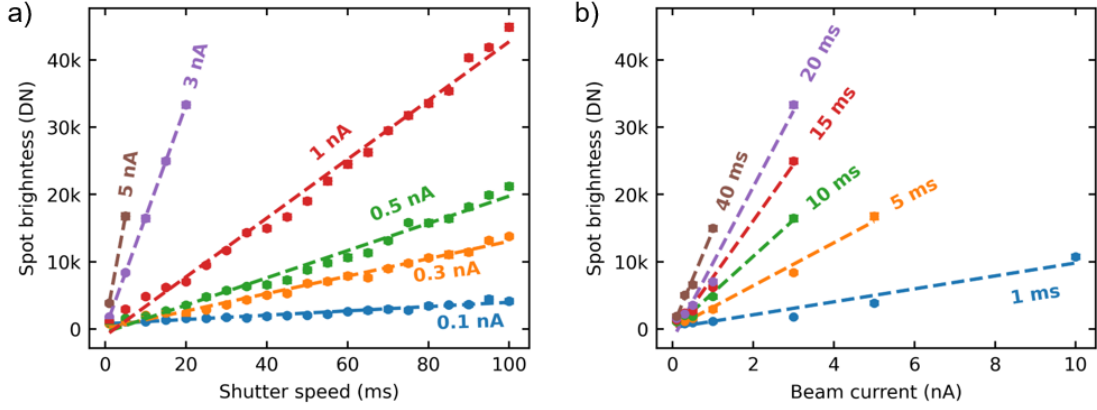


Figure 7.4 Measurement of SEM beam currents using the Raspberry Pi HQ camera. (a) shows the measured integral DN of the brightness spot generated by beams of different currents, measured at different shutter speeds. The plot proves the linearity of the sensor's response for electron detection. In b) the spot brightness is plotted versus the incoming beam current. The linear proportionality suggests the possibility of direct current calculation from the brightness. The plots are adapted from data published in [102] © 2023 IEEE.

Similar to photons in the visible spectrum, a linear relationship between measured brightness and exposure time of the camera can be established for the detection of electrons via presumably bremsstrahlung. Figure 7.4(b) additionally shows the spot brightness as a function of the beam currents for shutter speeds from 1 to 40 ms. This indicates that there is also a direct proportionality between incoming current and measured brightness. Thus, it would be theoretically possible to calculate the current directly based on the brightness. However, the sensitivity of the camera slightly varies over the chip, requiring very laborious calibration. Moreover, due to the energy-dependent X-ray photon transmission and yield, the ratio of brightness to incident current is expected to be accordingly dependent in a nonlinear manner. When measuring in diode setup, the electron energy is determined by the field emission extraction voltage. Therefore, a detailed energy dependent calibration of the sensor would be necessary. Instead, the relative proportions of the brightness distribution are used here to calculate the individual currents (see Chapter 7.3.1) for every measuring point, since here the extraction voltage and hence the electron energy is constant.

Another insight that can be gained from the measurement shown in Figure 7.4 is the confinement of useful shutter speed ranges. Currents exceeding 1 nA already lead to overexposure at shutter speeds beyond 20 ms. However, the current densities of these SEM beams are significantly higher than when measuring FEA currents due to their small spot size. Preliminary tests showed that the radii of the emission spots of single emitters of the FEA are sometimes three to four times larger than those of the SEM beams as a result of the divergence. Thus, the current densities here differ by a factor of 9 to 16. In order to cover the currents of the herein measured FEAs, ranging from approximately 100 pA to some hundreds

of nanoamperes (see Chapter 7.3), a distribution of four shutter speeds (31.5 ms, 10 ms, 3.15 ms and 1 ms) was found to be suitable.

7.2.3 Standard Measurement Setup and Procedure

The basic measurement setup is similar to that described in Chapter 4.2.1, but differs with respect to the sample holder and the distribution of the potentials. A schematic representation thereof is shown in Figure 7.5.

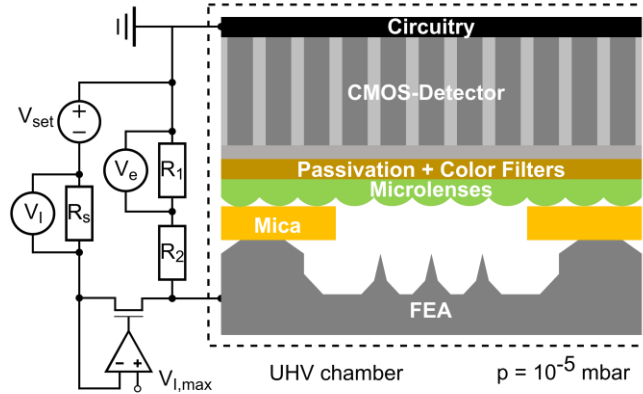


Figure 7.5 Schematic illustration of the measurement setup. Reprinted with adaptations from [232] with permission from the American Vacuum Society (AVS).

The FEA to be measured is placed face down on top of the camera, with a mica sheet sandwiched in between for electrical insulation. If not stated otherwise, the mica sheet has a thickness of approx. 60 μm for all measurements shown here, in order to avoid damaging leakage currents to the camera even under poor vacuum conditions and possible outgassing of the camera. The whole stack is clamped by an aluminum frame, which contacts the FEA and is fixed by two PEEK screws to corresponding holes in the PCB board of the camera. Before carrying out a measurement, sufficient evacuation of the vacuum chamber should be ensured in order to account for the outgassing of the PCB. The camera is controlled via a Raspberry Pi 4, which is placed in a ventilated housing outside the vacuum chamber. It is connected to the camera via two flexible ribbon cables and a vacuum feed-trough and communicates with the measuring PC via WLAN.

For the measurements a diode configuration is used, where the camera acts as the extraction anode. To ensure the functionality of the readout circuitry the camera is set to ground potential. Therefore, the FEA is pulled to a negative potential, which is provided by a high voltage power supply from the current control circuit [185]. During current regulation the FEA's potential can differ from the set voltage. Therefore, a high-resistive voltage divider is used to measure the actual voltage applied to the FEA. Unlike for the circuit described in

Chapter 4.2.2, it is not possible to measure the FEA current directly with a multimeter, because of the high voltage potential. Instead, the emission current is measured voltage-free by measuring the current passing a shunt resistor with an instrumentation amplifier. It is then transferred to the low-voltage side by an isolation amplifier and measured with a multimeter. The maximum extraction voltage of the setup is limited to 1.4 kV due to the herein used MOSFET. Empirical tests showed that high voltages can lead to excessive vacuum arcing, which disrupt the communication of the camera and in the worst case cause a defect of the sensor. Therefore, all measurements shown here are performed with a maximum voltage of 1 kV, if not stated otherwise.

An important prerequisite for the measurement is the synchronicity of the electrical and optical data. The control of the peripheral devices for electrical measurement is done by a microcontroller, while the camera is controlled via the Raspberry Pi. In order to synchronize those two a socket network is used. In this, the Raspberry Pi acts as a server and the measurement PC serves as client. As soon as the measurement starts, the PC transfers the desired electrical set parameters to the microcontroller, which configures the peripherals accordingly. Then, the PC sends a string via ssh to the listening server (Raspberry Pi), which in turn initiates an image acquisition routine according to the parameters passed in the string. Once the routine is complete, the Raspberry Pi sends a string to the PC, prompting it to request the desired measurement data from the peripherals. In current-controlled measurements, this is the extraction voltage necessary to achieve the specified current as defined by the regulation circuit. That way, a synchronous recording of all relevant measurement parameters is ensured. Unless otherwise mentioned, three images are taken for each of the four shutter speeds (31.5 ms, 10 ms, 3.15 ms and 1 ms) resulting in a total of 12 images per measurement point for all measurements shown here. Due to the approximately four to six seconds needed to capture the 12 images, each measurement point is repeated once to account for possible degradation during the acquisition by statistic evaluation. Figure 7.6 provides a schematic representation of the data acquisition cycle for current controlled measurements.

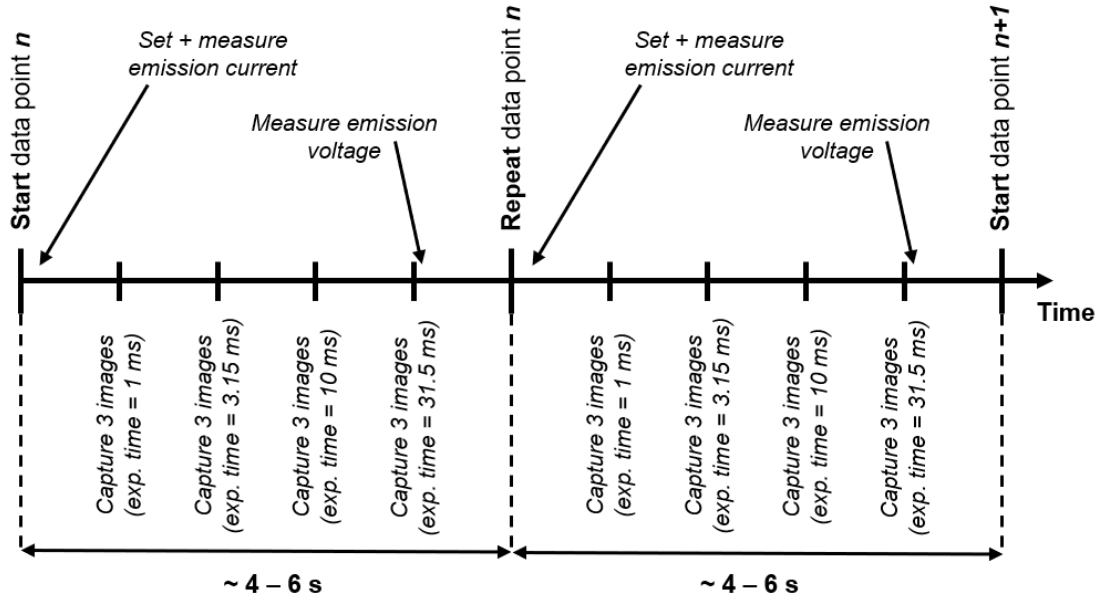


Figure 7.6 Timeline of the data acquisition procedure for current controlled measurements. Each data point is repeated twice to account for the 4–6 s needed to obtain the image data.

7.2.4 Image Post-Processing

As mentioned in Chapter 7.2.2, the measurement generates about 1 MB of data per second. Depending on the type and duration of the measurement, this can quickly add up to several GB of image data. The goal of post-processing is therefore to statistically evaluate the large amounts of image data and convert them into simple text documents, with sizes below 100 kB, containing all relevant data for further investigations.

The largest part of the data is due to the use of multiple shutter speeds, whose necessity is illustrated by the example measurement shown in Figure 7.7. Figure 7.7(a) depicts an SEM image of a single tip of the measured FEA and Figure 7.7(b) an overview of the whole array in top view, respectively. The white dashed circle here symbolizes the opening of the mica sheet, which is 30 μm thick in this case. Figure 7.7(c, d) represent images that were captured by the camera during the measurement with a shutter speed of 1 ms and 10 ms. The FEA was emitting a current of 4.4 μA at an extraction voltage of 1.2 kV at this data point. The white circles in Figure 7.7(c) mark the emission spots that could be detected for both shutter speeds. The green circles in Figure 7.7(d) show the emissions that could only be detected with the higher shutter speed and the associated increase in integration time of the signal. At the same time, the higher exposure time also leads to more saturation of pixels in the emission spots, which is marked here in red. The resulting loss of information can be circumvented, thanks to the linearity of the sensor, which was shown in Chapter 7.2.2. By linearly extrapolating the corresponding spot brightness captured at lower exposure time without any pixel saturation, the saturation-free brightness at the highest shutter speed can be reconstructed. This way, a

composite image with an almost arbitrarily expandable dynamic range can be generated by combining the information from multiple shutter speed images. Since entire spots with saturated pixels are replaced for this purpose, the basic prerequisite for this is reliable detection of the individual emission spots.

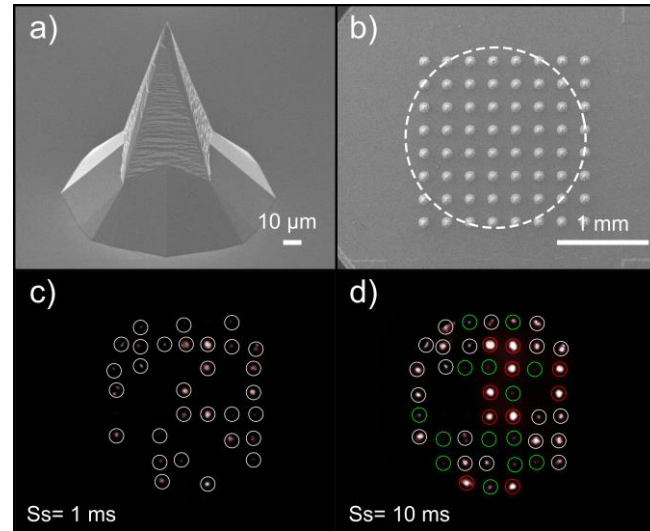


Figure 7.7 Example for a field emission imaging measurement using the Raspberry Pi HQ camera. In (a, b) SEM images of the measured TMAH FEA are depicted. The dashed line in b) indicates the position of the mica sheet opening. (c, d) are images captured by the camera during the measurement at shutter speeds of 1 ms and 10 ms, respectively. Emission spots with saturated pixels are marked with red circles. The green circles in (d) indicate spots that could not be measured for the shorter exposure time in (c). Reproduced with adaptations from [232] with permission from the American Vacuum Society (AVS).

All post-processing was programmed in a Python environment. In order to bundle all the necessary information of an electrical measuring point in one image, all related individual images must first be statistically evaluated together. Due to the three images per shutter speed and measuring twice for each current level, there are six electrically related images per shutter speed and data point. The algorithm evaluates each of these data points one after the other. In the first step, the six images of the data point are read in and averaged into one 16-bit array, containing the averaged gray values of every pixel. Their corresponding uncertainty is calculated by empirical standard deviation and is stored in a second array. Subsequently, the dark images taken at the beginning of each measurement are analogously averaged for the respective shutter speed and subtracted from the averaged array. The combined uncertainty of the resulting image is passed on according to the Gaussian error propagation via addition in quadrature of the individual errors of the dark image and measurement image. This results in one averaged array of gray values for each pixel including their uncertainty for each shutter speed of every data point in the measurement.

The next step is automatic spot detection for each averaged image per shutter speed of a data point. For this purpose, an algorithm based on the OpenCV library was developed. This

library provides a variety of implemented functions for processing the acquired emission images. It should be noted that OpenCV can only work with 8-bit image data. Therefore, the 16-bit images to be processed here are transformed by removing the 8 least significant bits. Subsequently, the image must be binarized by thresholding. The choice of the correct threshold value is crucial here to be able to distinguish pixel noise from detected emission. In the first iteration, this is determined by the algorithm proposed by *Otsu* [274]. Then, the binarized image is run through an edge detection algorithm according to *Suzuki and Abe* [275]. This detects the outer edges of connected structures and returns their coordinates. Finally, the center coordinates of the outer edges are determined and stored in a list. Due to the already mentioned emission current differences of several orders of magnitude and the associated variation of brightness between the individual spots, it is often not possible to find a perfect threshold that images both very bright and very dark points at the same time. In order to ensure that no spot remains undetected, the spot detection algorithm subsequently iterates through all possible 8-bit threshold values again in steps of five and adds the returned center coordinates to the list.

The resulting list of center coordinates now contains many falsely detected spots that must be sorted out. For this purpose, a readout radius is passed to the algorithm, which usually corresponds to half the distance between two tips in the FEA. The total spot brightnesses are determined using the untransformed 16-bit images. With this, the accumulated gray values of all detected spots are read out within this radius around every detected center coordinate. If the readout areas of two listed centers overlap, a middle coordinate of these two is determined and the brightness of this new spot is evaluated. If this is brighter than that of the two initially overlapping spots, the new center point coordinate is passed to the final list. Otherwise, the brighter of the two original spots is used. Spots with an accumulated brightness below 500 DN are declared as noise and are not considered in the final list. Spots with at least one overexposed pixel receive 100% uncertainty, in order to be identified later. If there is no saturation in the spot, the measurement uncertainty of an emission spot is calculated according to Gaussian error propagation via addition in quadrature of the individual uncertainties of each pixel. The total brightness is calculated by summing all pixels above 255 DN, pixels with 255 DN and below get 100% uncertainty, since they are suspected to be noise. The total brightness is needed later to calculate the individual tip currents. Again, the error propagation is done by Pythagorean addition of the single errors.

After going through this procedure for each individual data point, the results are saved in a text file for each shutter speed. In this file, all relevant information is stored line by line for each data point. Finally, the last step of post-processing is the extrapolation of the image

data. For this purpose, the text files are read into a Python programming environment again. Starting from the file with the highest shutter speed, each measuring point is then checked for spots with saturation, recognizable by an uncertainty of 100%. If this is the case, the file with the next lowest shutter speed is searched to see whether a corresponding spot was detected here as well. For this purpose, a center coordinate in the range of the readout radius around the center of the spot to be replaced is searched for in the respective data point. If this also shows saturation or no corresponding coordinate can be found, the text file of the next lower exposure time is searched. Otherwise, the brightness of the spot measured with lower integration time is extrapolated linearly according to the quotient of both shutter speeds and the spot with saturation is replaced. If no suitable replacement spot without overexposure can be found, the original spot retains its 100% uncertainty. Again, the results are saved in an analog text file. This includes all the electrical measured values (set voltage, extraction voltage, emission current, chamber pressure), the information about the individual spots obtained from the evaluation and extrapolation of the images (center coordinate, spot brightness), as well as the integral image data (integral pixel brightness, integral brightness of all detected spots), along with all associated uncertainties.

7.3 Analysis Methods for the Emission Behavior of Field Emitter Arrays

In this chapter, the unique insights into the emission behavior of FEAs provided by the QFEI measurement method are further explored through a test measurement of a TMAH FEA (see Chapter 5.1). Various types of analysis methods are presented and critically discussed to explore the capabilities of this measurement and to establish a toolbox for data evaluation. Note, that the presented methodology has already been used in a published study [103], which will be discussed in detail in the subsequent chapter. The herein measured sample was fabricated from a phosphorus doped <100> silicon wafer with a resistivity of 10–20 Ωcm . The outer dimensions of the die were reduced to 4 mm x 6 mm to allow the FEA to fit on the sensor surface. An array of 30 x 30 individual tips with a pitch of 66.7 μm on a total area of 2 mm x 2 mm is located in the center of the chip. The average height of the tips is about 30 μm , although there may be geometric differences within the array due to the wet chemical etching step. Figure 7.8 shows an SEM image of the entire FEA. The insets depict magnified views of selected individual tips to illustrate the minor geometric variations within the array. Due to the manufacturing process, the tip apexes are about 70 μm embedded relative to the

surface of the chip. Together with the 60 μm thick mica sheet, this results in a distance from the sensor surface of 180 μm .

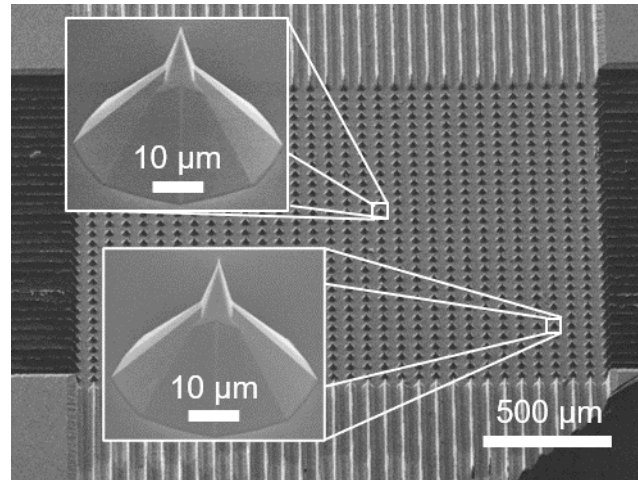


Figure 7.8 SEM image of the FEA under study. The wafer diced and TMAH etched array consists of 30 x 30 tips. A random inspection of the individual tips revealed no significant differences in geometry, as can be seen from the insets depicting a magnified image of two selected tips in detail.

The measurement procedure of this FEA is based on the methodology of the current-dependent lifetime test described in Chapter 4.2.4. However, only one set current level of 1 μA is measured to become familiar with the characteristics of this novel measurement method. As usual, the measurement is divided into three parts. First, seven consecutive current-controlled IV sweeps (four up, three down) are performed on the unused FEA, ranging from 5 nA to 1 μA . This is followed by a maximum one-hour constant current measurement at 1 μA . Hereby, the measurement control is programmed to stop the CCM and proceed to the next step when the control current is not reached within the available extraction voltage of 1 kV for ten consecutive data points. Finally, IV sweeps are repeated in a manner similar to the initial operation to analyze any changes in the characteristics due to the constant current load. The measurement was conducted in the setup schematically presented in Figure 7.5 at a chamber pressure of 10^{-5} mbar. Figure 7.9 demonstrates the data that can be obtained from this measurement using the evaluation methods described above. The left, middle, and right parts represent the initial sweeps, the CCM, and the subsequent sweeps, respectively. For sweeps, the x-axis describes the extraction voltage applied to the FEA. For the CCM depicted in the center, it indicates the elapsed time under constant current load operation. The integral emission current from the FEA is shown in red. The extraction voltage required to maintain 1 μA during the CCM is plotted in gray. In addition to these electrical measurement data, the integral brightness of all detected spots is shown here in blue, as well as their respective

number in green. The error bars for both the x- and y-directions were determined by calculating the standard deviation from the average of two measurements taken per data point.

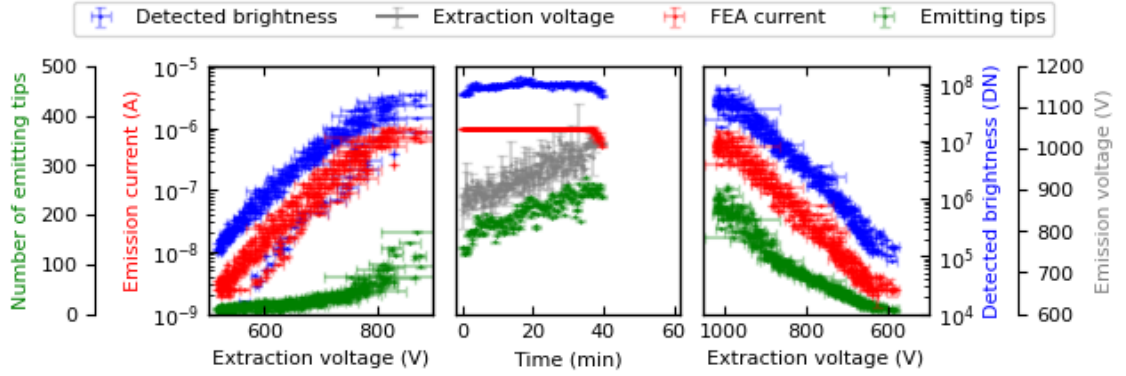


Figure 7.9 Raw measurement results for the n-type TMAH FEA. The measurement is divided into three parts: initial IV sweeps (on the left), a constant current measurement (in the middle) and final IV sweeps (on the right).

As anticipated, the brightness is largely consistent with the emission current. However, there is a slight but consistent increase in logarithmically plotted brightness during the CCM, which is likely due to the rising electron energy resulting from the linearly increasing extraction voltage. Furthermore, there are random fluctuations present that cannot be attributed to the emission current. These effects may be due to charging or changes in emission sites, which could impact the camera's sensitivity. However, these factors only vary by single digits, so they should not significantly affect the estimation of current magnitudes.

Regarding the general course of measurement, it is evident that the extraction voltage continuously increases during the CCM until it reaches the maximum of 1000 V after about 40 minutes. At this point, the continuous degradation of the FEA caused by the constant current load leads to the collapse of the emission current due to the limited voltage and thus to the termination of the CCM. According to the literature, this device failure is typically explained by a limited number of particularly sharp tips carrying most of the current load [143], [228], [276]. As these tips degrade, the voltage increases, causing a subset of less sharp tips to take over until there are eventually not enough tips remaining to carry the control current or the voltage limit of the measurement is reached. This explanation is supported by the number of emitting tips, which increases continuously with the voltage. In this and the subsequent chapter, we will analyze the current distribution in the array quantitatively to determine if this explanation fully explains the situation.

7.3.1 Individual Tip Emission

By combining the image data with the electrical data, the detected spot gray values can be translated into currents. For this purpose, the integrally measured emission current is distributed relative to the brightness shares of the detected emission spots for each measurement point, according to

$$I_{Tip} = \frac{I_{FEA}}{B_{FEA}} \cdot B_{Spot} , \quad (7.2)$$

where I_{FEA} is the emission current, B_{FEA} is the accumulated gray value of all pixels of the sensor, B_{Spot} is the respective spot brightness and I_{Tip} is the current of the correspondingly emitting tip.

At this point it should be emphasized that this approach presents a simplified version of reality for several reasons. As mentioned above, the measured brightness in the sensor is influenced by the extraction voltage dependent electron energy, which is related to the photon yield, the transmission rate, and the generated charge carriers. Due to the repeated measurement of each data point and subsequent averaging of the electrical data for each image, the extraction voltage can be considered constant for this calculation at a macroscopic level. Microscopically, however, possible local voltage drops in the tips must be taken into account, especially in the case of semiconducting FEAs. These can occur, for example, due to the depletion region in the case of saturated emission (see Chapter 2.3). Simulations showed that this voltage drop can be in the range of 75 V for p-doped FEAs with the geometries and extraction voltages studied here [190]. Furthermore, many different effects can interfere with or change the signal generation during the measurement. For example, there could be Coulomb interactions due to charging effects of non-conducting layers, changes in transmission rates due to material deposition or ablation on the sensor surface, or variations in sensitivity between sensor pixels due to manufacturing, to name a few. The extent to which these effects influence the reliability of the measured values has not yet been conclusively investigated due to a lack of verification options. However, all observations suggest that they are only of secondary importance, as the measured results are typically in good agreement with the literature. Furthermore, all data shown here are based on statistics and relative before-and-after comparisons to account for possibly large measurement uncertainties.

Going back to the measurement, the data obtained permits an analysis of the spatial distribution within the array to be displayed for individual each data point. Figure 7.10 illustrates a histogram and corresponding heatmap of the current distribution in the FEA at the beginning (Figure 7.10(a,b)) and at minute 37 (Figure 7.10(c,d)) of the CCM. This is when

the integral current begins to decrease and no longer reaches the control current (see Figure 7.9). To ensure comparability, the same bin edges were used for both histograms. They were selected by applying the Freedman-Diaconis rule [277] to the distribution at the beginning of the CCM.

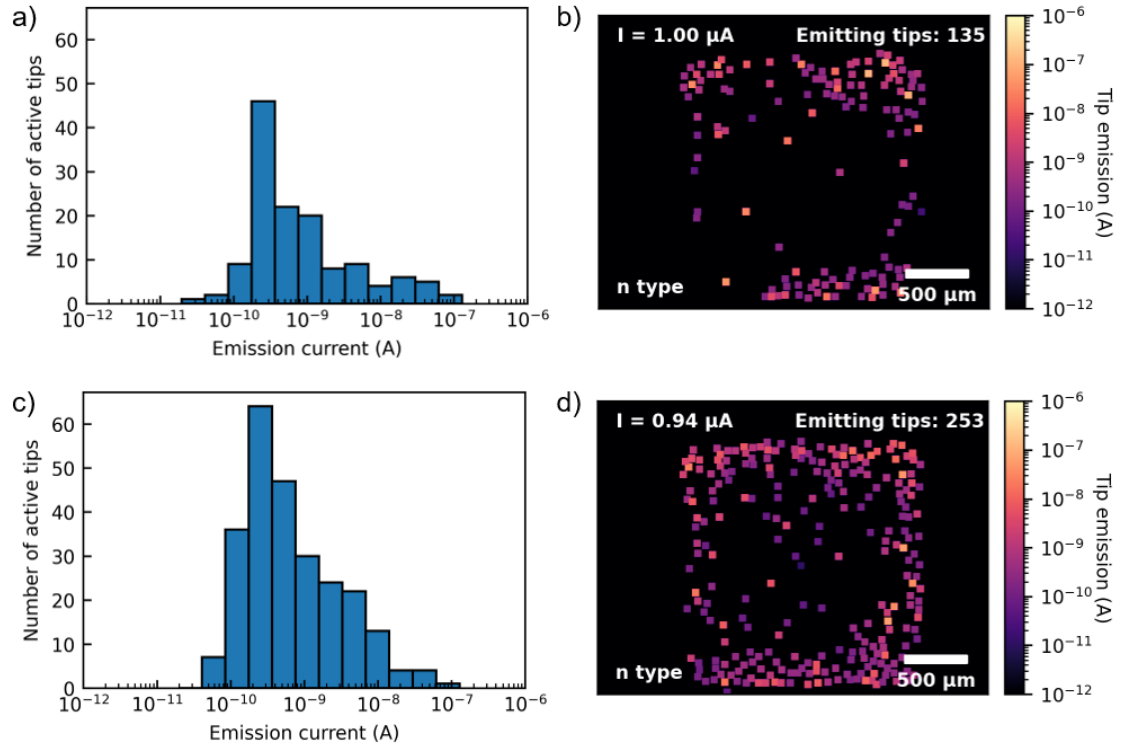


Figure 7.10 Analysis of the current distribution in the array at the beginning (a,b) and end of the CCM (c,d). (a,c) shows the two histograms for the quantitative analysis and (b,d) the corresponding heat maps for the spatial evaluation.

It is known from the literature that the fabrication of nanometrically sharp emitter tips typically results in a lognormal distribution of tip radii [145], [228], [236], [276]. Our group has confirmed this trend for this particular FEA type by means of SEM analysis as well [3]. Additionally, sharper tips typically emit higher currents at a given electric field [228], [276]. Therefore, the distributions of individual tip emission currents in Figure 7.10(a,c) are heavily skewed towards lower currents, representing duller tips. Over the course of the CCM degradation of the individual tips occurs due to numerous reasons, as outlined in the introduction of this chapter. To compensate for this, the current regulation increases the extraction voltage, which leads to the activation of new tips. Therefore, the total number of emitting tips increases from 135 to 253 during the 40 minutes constant current emission. During this, the distribution narrows and shifts its maximum and center toward less strongly emitting tips.

The heat maps in Figure 7.10(b,d) show that the emission within the array is quite unevenly distributed, with the majority of emitting tips located at the edges of the FEA, despite

the negligible geometric differences between the individual tips noted during SEM evaluation. The most intense emitting tips are scattered throughout the array, albeit in small numbers. Note that at the end of the CCM different tips than at the beginning carry the most dominant fraction of the integral current, indicating enhanced degradation of the strongly emitting tips.

With these representations, the changes in the current distribution in the FEA can be studied in detail for each individual measurement point. However, at this point of the evaluation, all measuring points are still considered independently of each other. In order to assess the evolution of individual tip emissions, the spatial resolution of the measurement can be used to reconstruct the emission of each individual tip over the entire course of the integral operation. Another python-based evaluation is used for this purpose. Starting with the first measurement point, a list of tip coordinates is created based on the detected emission spot centers. For each subsequent measuring point, it is checked whether the occurring spots lie within the readout radius around one of the listed tips. If this is the case, the corresponding emission current is assigned to the tip. Otherwise, a new tip position is defined.

It should be mentioned here, that this method is prone to two types of errors. First, it assumes that each tip causes only one emission spot and that this spot ideally occurs directly above its center. This is only conditionally the case, since asymmetric tip geometries with possibly multiple microscopic apexes, as well as interactions of the electric field lines of neighboring tips can lead to offset and split emission images. Therefore, it must be ensured that the readout radius is large enough to accommodate these effects. Using the number of pixels that correspond to at least half of the tip spacing in the FEA turned out to be a reliable approach for this. In this case, the value was therefore set to six. Due to the reduced resolution (see Chapter 7.2.2), this is equivalent to 24 pixels with a diameter of $1.55\text{ }\mu\text{m}$ and thus a radius of $37.2\text{ }\mu\text{m}$ for a tip distance of $66.7\text{ }\mu\text{m}$. The second possible source of errors is the spot detection. Particularly but not exclusively low brightness spots can still be missed from the spot detection at individual measurement points, which can result in gaps in the emission data of the single tip. It is therefore advisable to provide sufficient data points for reconstruction, especially during repetitive sweeps. For this reason, the number of data points for the measurement shown here was set to 40 points per sweep. In the case of current controlled sweeps, each of these data points has an associated set current. Using this, the data points of the individual emitter characteristics can be assigned to each other and averaged in current and voltage. Thereby, an uncertainty based on the empirical standard deviation can be assigned to each measuring point again. In order not to underestimate this uncertainty due to a small data base, the error given by the pixel brightness is passed on in addition to the statistical

error by error propagation and addition in quadrature. During CCM, such averaging is not possible, which is why the fluctuations of the individual tips can be overestimated here due to errors in point detection and the aforementioned uncertainties of the measurement itself. However, the basic course should also be valid here, since these errors occur only rarely and the measurement uncertainties should be in the percent range and not orders of magnitudes of the reading, especially at the comparably low individual current levels investigated here.

Figure 7.11 shows the results of these evaluations for three selected single emitter characteristics together with the averaged integral electrical measurement data of the FEA. These emitters were chosen to represent the various types of individual tip degradation that may occur during and as a result of the CCM.

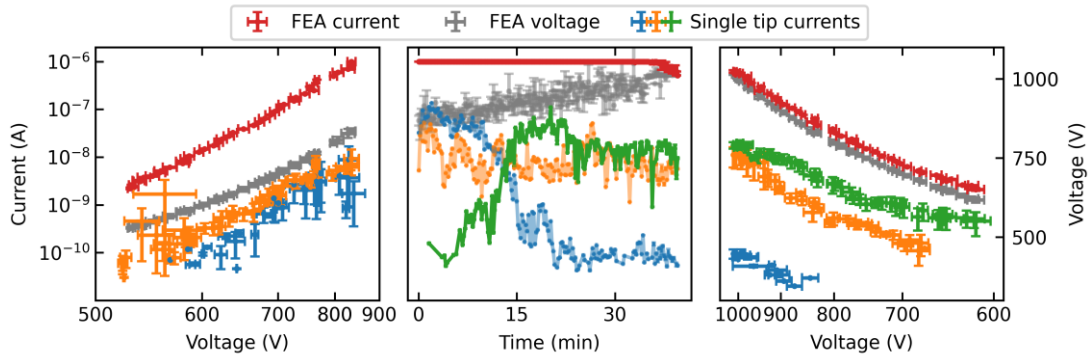


Figure 7.11 Averaged emission curves of the entire FEA and selected individual tips over the whole course of the measurement. The IV characteristics (on the left and right) are plotted in adapted ML coordinates, with reciprocal distances between the non-reciprocal voltages for better readability.

Based on these emission curves, we can determine the specific contribution of each tip to the total current at each point in time. Here we shall use this to analyze the possible emission patterns of the individual emitters. The tip plotted in orange started emitting at the very beginning of the measurement, with a turn-on voltage around 530 V during the initial sweeps. In the averaged characteristics it reaches a maximum current of about 10 nA. During CCM, it exhibits a behavior that would not be expected at first. Instead of reaching a maxim current, which subsequently leads to an excessive current density resulting in complete failure of the tip, it degrades repeatedly, whereby the emission is also frequently regenerated by the increasing extraction voltage. This can be the case, if the degradation is related to adsorbates. As already mentioned in Chapter 3.1, depending on the type of contamination, the emission can be both increased and decreased by changing the work function and emission probability [82]–[84]. Local changes in temperature or the ionization rate of the residual gas can impact the balance between adsorption and desorption. The contamination of the surface with adsorbates is therefore at least partly reversible [83]. On the other hand, thermal runaway [3],

[200], [201], [203], [204], [207]–[209] or ion bombardment [202], [203] can cause irreversible change in the tip's geometry. Due to the high chamber pressure during this measurement, a strong reversible influence of adsorbates is to be expected here. Mostly, however, strong emission leads to a mixture of reversible and irreversible degradation. Comparing the sweeps before and after the CCM, the orange emitter returned to about the same current level as before, but required about 150 V more extraction voltage. A completely different behavior can be observed for tips shown in green and blue. The IV characteristic of the blue tip before CCM is very unstable, as can be seen from the large error bars, indicating a slower activation of the tip. However, during the first minutes of CCM, it emits very strongly with a maximum current of over 100 nA. This appears to be leading to irreversible degradation, as the emission current continuously decreases from there until it reaches the low picoampere region. Interestingly, the tip never stops to emit, but does not significantly contribute to the integral current of the FEA anymore. The green tip, on the other hand, shows a complementary course. While it makes no contribution at all during the initial sweeps, it starts emitting during the first minutes of the CCM until it finally reaches a maximum current of over 100 nA around minute 20. This very late activation can have several causes, apart from the increasing extraction voltage. On the one hand, the CCM leads to a rising FEA temperature, since there are no cooling phases as during the sweeps. Therefore, the adsorbate contamination can decrease, leading to changes in work function, emission probability and effective extraction field at the apex of the tip [82], [84]. On the other hand, the incipient emission can cause further desorption or sharpening of the tip due to the ion bombardment [202], [203]. Despite the similar maximum current compared to the blue tip, there is only slight degradation present. As a result, the green emitter outperforms even the orange one in subsequent sweeps.

This detailed analysis of only three individual tips of the FEA gives an idea about the variety of partial contributions that can make up the integral emission current. However, the reasons whether and how emissions change are still numerous and intertwined. To discuss the interactions of all 900 potentially contributing emitters, a more statistical examination of the distribution is necessary.

7.3.2 Emission Distribution Analysis

With an ideal FEA, the current load would be evenly distributed among all tips in the array. This would minimize the expected degradation per tip. To evaluate how far a real emitter is from this ideal and how to approach it, the behavior of the current distribution in the array must be visualized in a clear and quantifiable manner over the entire course of the measurement. The challenge in illustrating this lies in the fact that the image data assigns an entire

current distribution, such as those shown in Figure 7.10, to each individual data point of the measurement. Figure 7.12(a) shows one way of illustrating this multivariate data in a two-dimensional manner. The current distribution obtained from the three-part measurement is presented here in the form of a triptych, similar to Figure 7.11 and Figure 7.9. The left section represents the initial characteristic, the middle section represents the CCM, and the right section represents the subsequent characteristic with a mirrored x-axis. Pseudocolor plots are used to represent entire distributions for each x-value. These work in a similar way to 2D histograms but allow pixels of varying heights and widths. The y-axis of the plots describes the emission current of the actively emitting individual tips. Therefore, the height of each pixel corresponds to the bin widths of a corresponding current distribution histogram. To ensure comparability, the bin edges in the y-direction must be fixed to the same values for each distribution and corresponding x-value. For all measurements shown in this work, this is done by spreading ten bins (or pixels in this case) per magnitude linearly on the logarithmic current axis. This provides a reasonable resolution for the herein investigated datasets. The width of the pixels in the x-direction is defined by the gap between two adjacent data points, which depends on the specific dataset. Unlike usual, the set current is plotted on the x-axis of the characteristic measurements instead of the extraction voltage. This is because the extraction voltage for one set current value can vary greatly from sweep to sweep, making it difficult to assign related data to a pixel. The set currents, on the other hand, are the same for all sweeps. Therefore, it is easy to assign the emission current of each actively emitting tip for a given integral set current to a specific pixel of the pseudocolor plot. Additionally, this method ensures a constant width of the pixels, as it follows the linear distribution of the set currents on the logarithmic current axis. The pixel's width of the CCM may vary slightly between different data points as it is determined by the time needed for data acquisition, which is typically around 4 to 6 seconds. The color of the pixels indicates the number of tips emitting in the corresponding current range at that point in time. Since the characteristics before and after the CCM each consist of a total of seven sub-sweeps, seven distributions are assigned to each x-value. Thus, the total number of assigned emission currents here outweighs those during the CCM. To ensure comparability of the distributions regardless of the data basis, the absolute values in Figure 7.12(a) were normalized with regard to the respective maximum count for each x-value. This also makes it easier to recognize the center of the distributions. For details regarding the absolute numbers, Figure 7.12(b) shows the number of simultaneously emitting tips together with the applied extraction voltage. Both the voltage and the number of tips are presented as individual sweeps in different colors in the case of the characteristic curve measurement. The order corresponds to that shown in the legend.

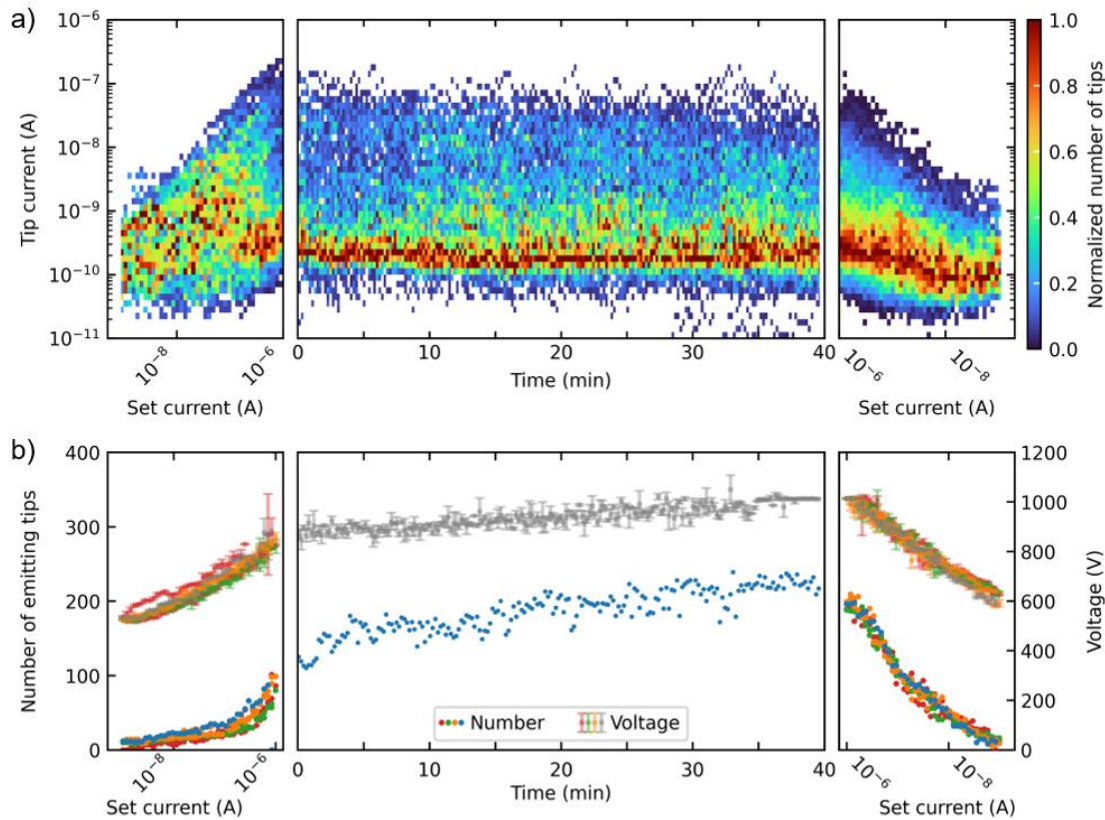


Figure 7.12 (a) Current distribution in the FEA over the entire course of the measurement represented in form of a pseudocolor plot. The colors were normalized for each x-value by the respective maximum count of the distribution. (b) Shows the absolute number of tips emitting simultaneously together with the extraction voltage applied to the FEA. The individual sweeps of the characteristic measurement are plotted in different colors. The order corresponds to that shown in the legend.

The resulting representation in Figure 7.12(a) reveals, that the current distribution during the initial sweeps is irregular and widely scattered. Figure 7.12(b) shows, that more and more emitters are activated with each sweep during this sub-measurement. Therefore, the fluctuating current distribution can be attributed to switch-on or conditioning effects of initially operated emitters [83], [187], [188]. The overlay of the seven sweeps also contributes to this. During the CCM, the weakest emitting tips are consistently represented in the highest number. This is in line with the literature's prediction that the current distribution in FEAs is typically skewed towards lower-emitting tips [143], [228], [276]. Overall, emission currents ranging from a few picoamperes to a few hundred nanoamperes can be observed here. However, the latter usually only occur for individual measurement points, as the corresponding high current densities quickly lead to degradation of the emitting tip. The number of tips emitting simultaneously continuously increases during the CCM, due to the increasing extraction voltage. After the CCM the distribution is less scattered than before, since most of the emitting tips have been sufficiently activated and particularly sharp tips are either dulled

or destroyed. The number of parallelly emitting tips does not change from sweep to sweep in this case.

An interesting aspect that can be further explored at this point relates to the increasing number of tips during CCM. It is known in literature, that a higher number of simultaneously emitting tips in FEAs typically leads to reduced fluctuation in the integral emission current during constant voltage operation [86], [278]. To examine how this applies to the emission stability of individual tips during constant current operation of the FEA, Figure 7.13 shows the averaged current fluctuation per tip over the course of the CCM. This fluctuation was calculated by accumulating the absolute difference between two adjacent tip current data points and dividing this by the larger of the two to avoid division by zero. Finally, the fluctuations of all tips were summed and divided by the number of active tips.

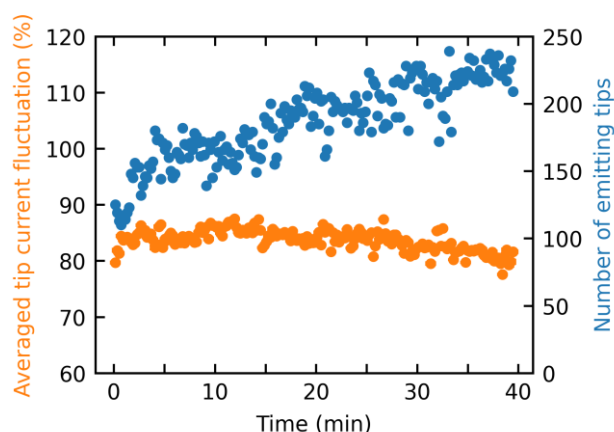


Figure 7.13 Average current fluctuation per tip during the CCM, compared to the number of actively emitting tips.

The figure shows a current variation percentage of the individual tips mostly ranging over between 80 and 90%. This high value may be attributed to uncertainties in the measurement method, as well as the influence of high chamber pressure and adsorbates. However, previous measurements from literature have shown that it is not uncommon for individual tips to experience current fluctuations exceeding 100%, even under better measuring conditions [278]. Therefore, this value is considered plausible. During the first three minutes, there is an increase in fluctuation visible in Figure 7.13, despite an also increasing number of emitting tips. This is likely due to the rising bulk temperature during the CCM, which affects the desorption rate of adsorbates and leads to variations in emission behavior. Until minute 15, the number of tips remains mostly constant, while the fluctuations still increase slightly. Starting at minute 15, there is a noticeable decrease in fluctuation combined with an increase in the number of emitting tips. This behavior is comparable to voltage-controlled measurements found in literature [86], [278].

The last aspect to be discussed concerns the way the respective tips contribute to the integral current. As it can be seen in Figure 7.12(a), most of the newly activated tips emit currents in the sub-nanoampere range. This is due to the fact, that sharper tips typically start to emit at lower voltages, while blunter tips are getting activated later, when the voltage increases further [143]. The total number of the sub-nanoampere emitting tips increases from 75 to over 150 over the 40 minutes of CCM. Therefore, they are accounting for a good two-thirds of all emitting tips at the end of the CCM. The emitters carrying over 1 nA of current show a flatter increase, which is why the mean current of the distribution is steadily decreasing. The formation of this stable base of low emitting tips also leads to a narrowing of the distribution. However, the decisive factor for the integral emission current of the FEA is, of course, the strongly emitting tips. The maxima in Figure 7.12(a) show tips emitting over 100 nA reoccurring throughout the whole measurement period, although they can typically only hold this current load for a few seconds. Analogous to the behavior of the orange tip shown in Figure 7.11, however, they are not necessarily destroyed by this, but degrade reversibly in part.

To better understand the role of each emitter group, Figure 7.14 shows the percentage of each bin's current to the total current of the FEA. This was obtained by accumulating the current of each tip within the specific bin and dividing it by the respective integral current of the data point.

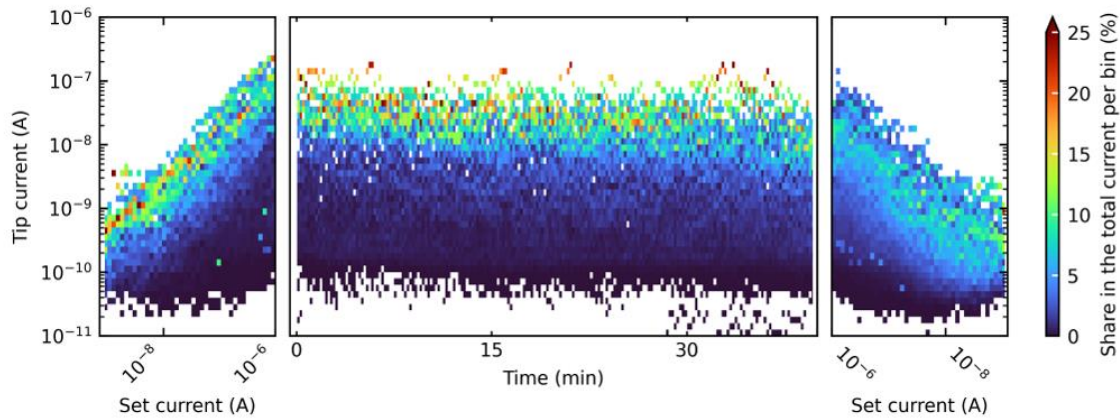


Figure 7.14 Center of the current distribution represented by the relative shares of each bin's total current.

Over the entire course of the measurement, the maximum of each distribution is consistently found at or close to the highest occupied bin. This aligns with the literature's predictions, indicating that the majority of the current is carried by a small number of highly emitting tips [143], [228], [276]. After the CCM, the distributions become flatter and broader, reducing the dominance of this behavior. This can be attributed to the degradation of most

highly emitting tips at this point, requiring more contribution from the less strongly emitting tips to the integral emission current.

In general, this type of analysis is particularly useful for characterizing changes in the current distribution during the CCM. However, due to the nature of distributions, only the totality of all active tips can be considered. The question remains as to what defines when these changes happen and how it can be controlled. A way of monitoring the changes of each individual tip while still allowing statistical evaluation, is provided by the analysis of the individual tip characteristics from the sweep data.

7.3.3 Characteristic Curve Analysis

There are two main parameters that can be extracted from the IV characteristics of field emitters, namely the FEF and the formal emission area. As previously noted, this area differs significantly from the actual surface area from which the electrons are emitting, as it was modeled for a planar ideal metallic emitter. In reality, this area depends on the voltage, work function and shape of the emitter [37], [195], [279], and therefore cannot provide easily interpretable values for characterizing individual emitting tips. Furthermore, it is calculated using the extracted FEF, which results in a strong correlation and therefore redundancy. The field enhancement factor on the other hand, provides a more appropriate quantity to describe the change of individual tip characteristics. As already mentioned, it corresponds to the amplification of the macroscopically applied field at the microscopic apex of the tip and is strongly related to the tip's radius of curvature, height and surface occupancy [33], [51], [53], [83], [96]. In contrast to the measurements shown so far, the QFEI allows for determination of the FEFs of each tip individually, thus avoiding errors caused by the superposition of multiple emitters. Therefore, the FEF is a promising candidate in this case to investigate the degradation phenomena happening during the CCM. As described in Chapter 2.4, it can be calculated using the slope of a linear regression of the characteristics in MG coordinates. Figure 7.15 illustrates the linearly fitted IV curves in MG coordinates of or a representative individual tip of the herein investigated FEA.

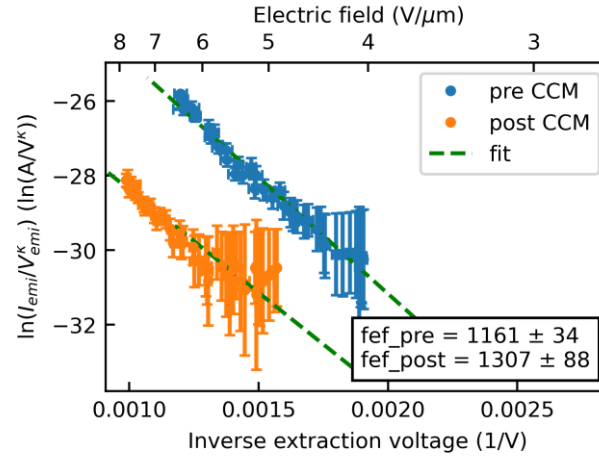


Figure 7.15 Extraction of the field enhancement factor from single tips by linear fitting in Murphy-Good coordinates.

In order to allow a statistical analysis of the FEFs in the array, this process was automated by incorporating it into the evaluation algorithm. Nevertheless, not all curves are appropriate for fitting due to possibly significant fluctuations or lack of data. Therefore, tips are excluded from this assessment if (1) there are fewer than 10 data points available, (2) the calculated uncertainty exceeds the FEF, (3) the uncertainty is greater than 500, (4) the FEF is negative, or (5) the χ^2 value of the linear regression exceeds the limit of 20 (refer to Chapter 2.4). The resulting distributions for the herein investigated FEA are shown in Figure 7.16(a,b) in histogram form. Figure 7.16(a) shows the FEFs of all assessable tips, and Figure 7.16(b) only displays those that were active both before and after CCM. In Figure 7.16(c), the respective spatial distribution in the array before and after CCM is depicted as a heatmap.

Consistent with the findings illustrated in Figure 7.10(b,d), the mappings presented in Figure 7.16(c) exhibit an uneven spatial distribution with a tendency towards higher FEFs in the array's peripheral areas. Nevertheless, there is no distinct hotspot of sharply pointed tips, which aligns with the results obtained from the SEM analysis (Figure 7.8). Examination of the histograms in Figure 7.16(a,b) reveals, that the log-normal distribution of the tip radii already mentioned above results in a positively skewed distribution of the FEFs. After activation during the CCM, the statistics improve. However, the form of both distributions remains largely unchanged and closely resembles the findings of *Nilsson et al.* in their examination of the FEF distribution in carbon thin films using scanning anode field emission microscopy [252]. It should be noted that the number of evaluable tips differs from the simultaneously emitting tips depicted in Figure 7.12(b), as not all emitting tips can be accurately fitted with linear regression. The mean FEF of all tips (see Figure 7.16(a)) decreased moderately while the standard deviation increased, due to the activation of high FEF emitters during the CCM. However, for the tips that were already active before the CCM, there is a slight

increase in the average FEF and a decrease in the standard deviation, indicating a narrowing of the distribution.

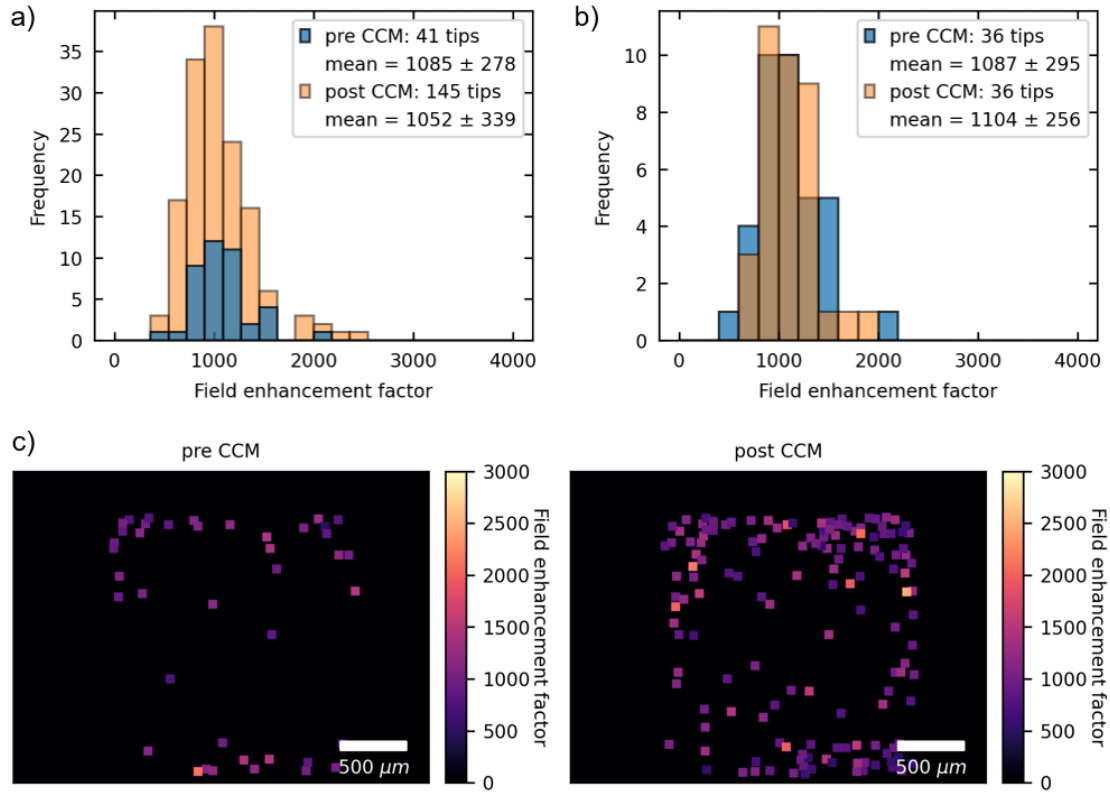


Figure 7.16 Field enhancement factor distributions in the array. (a) shows the FEFs of all tips in the array before and after the CCM, while (b) only illustrates the FEFs of tips that were active both times. In (c) the spatial distributions are shown as heat maps before and after the CCM.

To provide additional insight, Figure 7.17 illustrates the alterations in all FEFs that were assessed. The data in (a) is categorized into newly activated tips (grey), those deactivated during CCM (red), and those that remained active both before and after CCM (orange). In (b), a heat map displays the spatial distribution, with newly activated tips represented by a plus sign, deactivation by an x, improvement in green, and deterioration in red color. It is worth noting that there is no significant deactivation of tips during CCM. This can be attributed to the continuous increase in voltage as a result of current regulation, and a moderate integral emission current, which prevents the complete breakdown of the tips. The FEFs of the deactivated tips show an average of only 1071, indicating that excessive emission currents are not to be expected here. Considering the deactivated tips' close proximity to other emitters in Figure 7.17(b) there is even a possibility that the evaluation algorithm incorrectly assigned measurement data in this case.

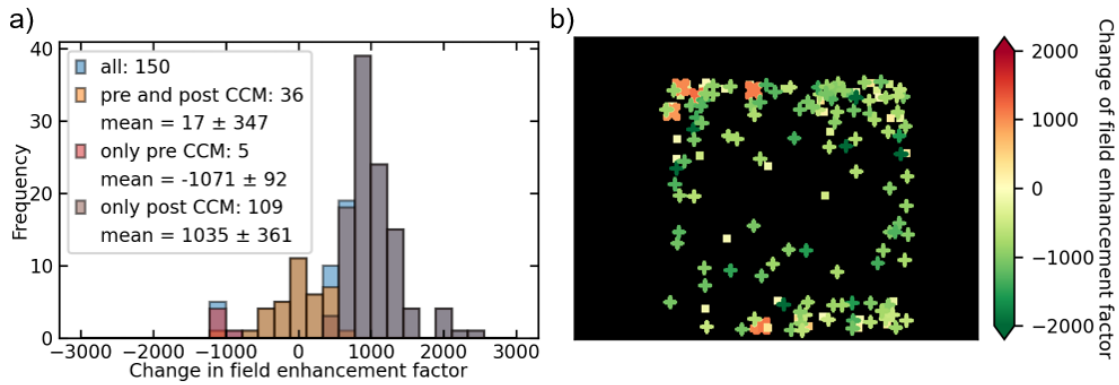


Figure 7.17 Change of the FEF due to the CCM depicted (a) as a histogram and (b) in form of a mapping.

The newly activated tips have a similar mean FEF, but are much more frequent with 109 compared to only 5 deactivated tips. The standard deviation of the mean value indicates a wider distribution of FEFs, possibly due to later activation and ongoing conditioning effects. The continuously active tips display minimal average changes. However, the distribution's width shows that significant changes must have occurred here.

To analyze this in greater detail, Figure 7.18(a) shows the FEF change for each tip as a function of its initial FEF before CCM. Tips that were disabled during CCM are marked in red, while those that remained active are in blue. The tips that were active at both times are fitted by linear regression, shown in black. Their mean change and initial FEF, including the respective uncertainty as an indication of the spread of the distribution, is plotted in brown. Supplementary, Figure 7.18(b) presents a graph comparing the FEF of each tip post CCM to its initial FEF. The green color represents tips whose FEF has increased due to the CCM, and the orange color represents tips whose FEF has decreased. Additionally, newly activated tips (blue) are positioned at $x = 0$ and deactivated ones (red) are at $y = 0$. The Figure includes the number of tips in each category, as well as a mean FEF before and after the CCM, similar to Figure 7.18(a). As previously stated, it is recommended to evaluate the depicted values in relation to each other rather than solely relying on their absolute values.

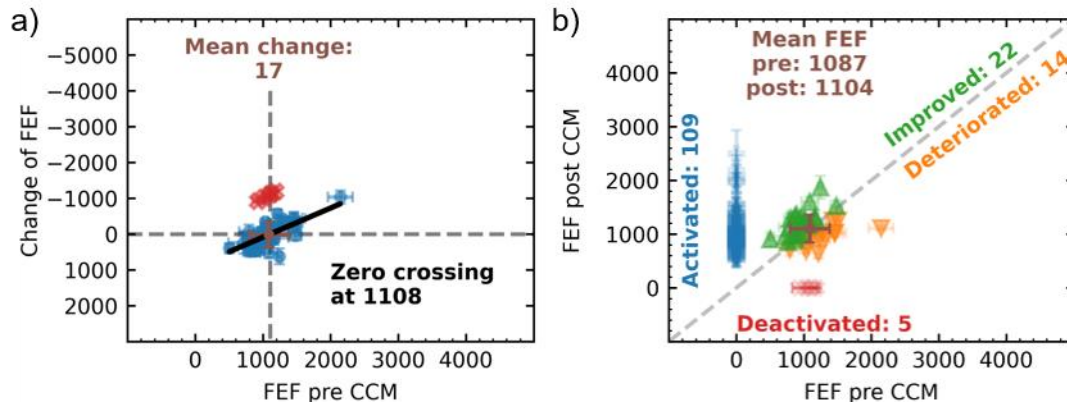


Figure 7.18 (a) Change of FEF as function of the initial FEF of each tip. (b) FEF pre CCM vs. FEF post CCM.

As expected, Figure 7.18(a) shows a linear correlation between initial FEF and tip degradation. Based on the linear regression analysis, a zero crossing can be observed at an FEF of 1108. Since the graph displays the change relative to the initial FEF, this value can be interpreted as a threshold FEF above which tips tend to deteriorate rather than improve. It is noteworthy that this value aligns nearly congruent with the mean FEF post CCM demonstrated in Figure 7.18(b). This suggests a possible burn-in process, that trims the individual tips of the FEA towards an ideal FEF for the specific operational conditions. However, further investigations are necessary to exclude coincidences as the mean change of FEFs in this measurement is close to zero (see Chapter 7.4). Based on Figure 7.18(b), it is evident that the marginal alteration in the mean FEF of the FEA is a consequence of the CCM's ability to both improve and degrade individual tip FEFs in the array. The ratio even inclines towards improvement in this case.

However, it is important to note that besides the general issues addressed in Chapter 3.1 the interpretation of FEFs must be approached carefully. Besides the problems that might originate from the interpretation of optical data, this factor is solely derived from the slope of the respective characteristic and disregards the absolute voltage or field required for emitting a specific current. In addition, despite implementing measures to prevent poor fitting (as outlined earlier in this chapter), there is still a possibility of errors. Especially outliers with very high FEFs can often be traced back to non-ideal tip characteristics.

An alternative approach, which is not based on fitting and provides information about the actual emission current of the individual tips, is shown in Figure 7.19. Here, the emitted current for each active tip was analyzed at the maximum integral set current of 1 μA , and the currents from each partial sweep were averaged for every tip. Figure 7.19(a) presents the respective tip current after the CCM plotted against its initial value including the standard deviation of the averaging. The plot's color scheme, follows the pattern previously shown in

Figure 7.18(b). Figure 7.19(b) shows the corresponding percentual change in the current of each tip that was active both times, represented in logarithmic form.

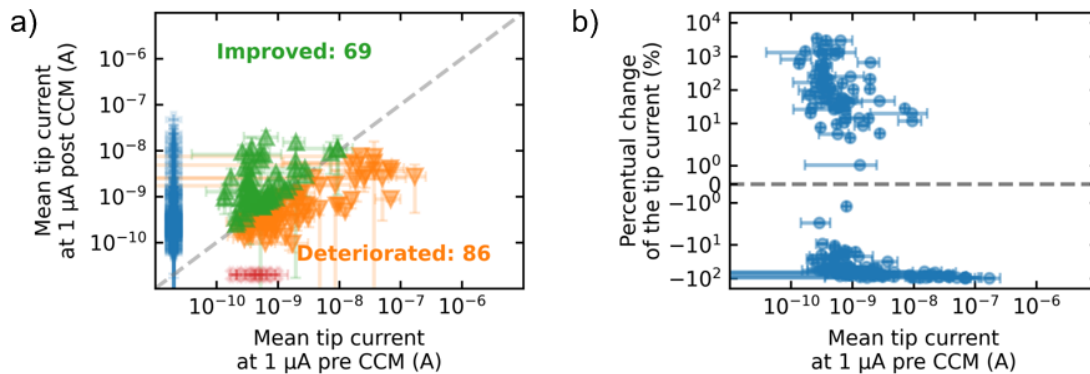


Figure 7.19 Comparison of the mean currents of each tip at the maximum integral FEA set current of 1 μ A during sweeps before and after the CCM. (a) plots the tip current after the CCM against its initial value. (b) illustrates the percentage change in relation to the tip current before the CCM.

Similar to the behavior of field enhancement factors, high levels of emitted current typically cause more pronounced degradation. Currents that exceed approximately 10 nA usually degrade during the CCM. Improvement and deterioration can both be observed below this value. Notably, Figure 7.19(a) demonstrates maximum currents are emitted after the CCM from newly activated tips and/or formerly low-current tips that improved over the course of the measurement. Many of the previously dominant tips emit in a range of 1 nA to 10 nA after the CCM. This range appears to allow stable operation for this FEA type and the specified measurement conditions. The total number of improved and degraded tips is subject to a high uncertainty because it is dominated by the high density of data points near the separation line. Despite this, the ratio of nearly one suggests that both improvement and degradation play equally essential roles in the array.

In summary, this section's results demonstrate that a comparative analysis of the individual tip's characteristics before and after the CCM, particularly regarding changes in the FEFs and maximum tip currents, can provide promising insights into the emission and degradation behavior of FEAs. The majority of the results are in good agreement with the literature. However, new findings are also emerging, such as the potential burn-in process indicated in Figure 7.18. This and other phenomena will be investigated in more detail in the following chapter.

7.4 Study of the Doping-Dependent Emission Behavior of Silicon Field Emitter Arrays

This chapter is an adapted version of my journal article [103] published in *Micromachines*, vol. 14, no. 11, p. 2008, Oct. 2023, doi: 10.3390/mi14112008. Discussions and figures are adapted and reprinted under CC BY 4.0 license.

In Chapter 7.2, the basic methodology for data acquisition and processing was explained. The previous chapter established the evaluation toolbox provided by the QFEI. In this chapter, the method is actively applied to investigate the influence of doping types and concentrations on the emission behavior of TMAH FEAs. Multiple samples were fabricated analogous to the fabrication process described in the beginning of Chapter 7.3. Three different substrate materials were used: lowly n-doped (phosphorus, resistivity of 10–20 Ωcm), highly n^{++} -doped (antimony, resistivity of $<0.005 \Omega\text{cm}$) and lowly p-doped (boron, resistivity of 10–20 Ωcm) silicon $\langle 100 \rangle$ wafers. It is important to note that the dopant type of the substrate can affect the orientation-dependent etch rate and selectivity of the silicon crystal planes in TMAH [280]. To achieve similar morphologies for the different doping types, the etching time for each sample was determined by sight. It was not possible to fabricate highly p-doped TMAH FEAs with comparable morphology to the other dopant types due to the fact that heavily boron-doped silicon acts as an etch-stop for TMAH [280]. SEM analysis of the fabricated samples confirmed the success of the process adaptation. All types have an embedding depth of 50 μm . Therefore, with a mica thickness of 60 μm , the distance between the camera and emitter tip is 110 μm . The electrical measurements were conducted using the setup described in Chapter 7.2.3 at a base pressure of 10^{-5} mbar. The measurement procedure follows the methodology outlined in Chapter 7.3. Seven current-controlled IV sweeps (four up, three down) ranging from 5 nA to 1 μA are conducted, followed by a maximum one-hour CCM at 1 μA . If a measured FEA does not achieve the control current for ten consecutive measurement points within the available extraction voltage of 1 kV, the CCM is programmed to terminate. Finally, another IV sweep measurement, similar to the initial one, is performed.

7.4.1 Integral Measurements and Single-Tip Emissions

Figure 7.20 shows the results of the measurements for three differently doped but morphologically similar FEAs. The sweeps before and after CCM (left and right) are plotted in adapted ML coordinates, where the x-axis has been relabeled and rescaled with reciprocal distances to show the voltage instead of its inverse. Additionally, the x-axis of the left part has been mirrored. In addition to the integral current (red) and the integral voltage (gray),

Figure 7.20 also shows the characteristics of three individual tips for each FEA (blue, orange, and green). These tips were selected to provide a comprehensive representation of the diverse range of current courses observed at individual emission sites. Note that the n^{++} -type sample's CCM was terminated prematurely because it could no longer emit 1 μA within the given extraction voltage limit of 1 kV.

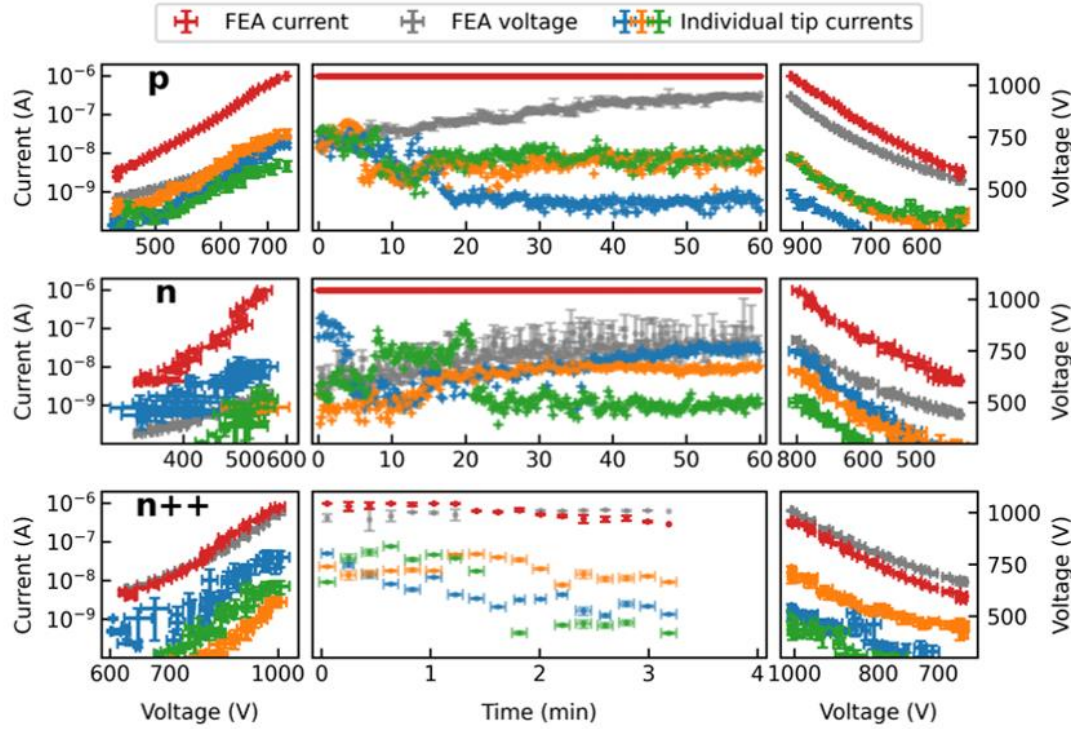


Figure 7.20 Measurement results for three differently doped but morphologically similar FEAs including the emission currents of three selected individual tips of each FEA obtained from the optical data. The IV characteristics (on the left and right) are plotted in adapted ML coordinates with the left x-axis mirrored. Reprinted from [103]. Licensed under a Creative Commons Attribution 4.0 (International License: <https://creativecommons.org/licenses/by-nc-nd/4.0/>).

The measurement of the p-doped emitter is presented in the top row of Figure 7.20. For many of this sample's individual tip characteristics, only three of which are shown here, clear saturation behavior can be observed prior and post to the CCM. As mentioned previously, the level of the current saturation hereby strongly depends on the charge carrier generation in the depletion region at the surface, which can be influenced e.g., by thermal or photonic excitation [70], [75]. In the case of this measurement, the expected strongly pronounced adsorbate coverage of the tips due to the high pressure in the vacuum chamber can also have a large effect on this value [190]. Additionally, it has been shown that geometric differences can influence current saturation, which might also be the case here [75]. For the herein-measured p-doped FEA, the saturation level can be mostly observed in the range of 1 to 10 nA. However, for the reasons mentioned above, saturation can also be significantly higher at times

(see orange and blue curves). At the beginning of the CCM, the FEA temperature increases due to the constant current load, which in turn favors the desorption of adsorbates. In the case of the orange and green tips, this leads to the recovery of the stable emission level in the range of 10 nA. In some cases, on the other hand, the excessive emission current also leads to irreversible degradation, as can be seen from the tip plotted in blue.

A similar behavior can be observed for the n-doped FEA. Emission currents at or below 10 nA usually do not cause significant degradation to the tip. Overly high emission currents result in either severe degradation (green tip) or burn-in to a constant emission level (blue tip). However, the orange graph shows that the progressive current load during the CCM does not necessarily lead to a deterioration of the individual tips. In fact, for all FEAs, many tips can be identified that improve over the course of the measurement and, in some cases, despite being activated later, can still contribute a relevant proportion of the total current. This will be further discussed in detail in Section 7.4.3.

Apart from this, it is noticeable from Figure 2 that the highly doped n^{++} -type FEA only reached the control current for about 3 min during the CCM, while the p-type and the lowly doped n-type FEA were able to emit for the whole hour at 1 μ A and 10^{-5} mbar within the available extraction voltage range. The highly n-doped FEA barely reached the integral control current during the sweeps and started the CCM with an extraction voltage of 1 kV already. On the one hand, this may be due to morphological differences, which could not be seen during SEM analysis. On the other hand, too much degradation during the initial sweeps may also be the reason. This will be discussed in more detail in Section 7.4.2.

An intriguing phenomenon has been increasingly observed in the case of n^{++} - and n-doped FEAs. By analyzing image data over the course of the CCM, it is apparent that strong emitting tips often activate the emission of their surrounding tips. This effect can be illustrated with Figure 7.21(a), which displays the emission heat map from minute 13 of the CCM of the n-type FEA. The markers' color reflects the emitted current of each active tip of the array. Their size corresponds to the area that is integrated when reading the distinct emission spot's gray values. The area where clustering occurs is marked with a red box. In Figure 7.21(b), the red line represents the current of the dominant tip (identified as the yellow emission spot in the heatmap) over the first 20 min of the CCM. The blue line indicates the number of actively emitting tips within the box during this time. The currents of other tips in the region are displayed as dots in multiple colors. A correlation between the current increase in the dominant tip to the number of active tips within the box can be observed. Similar behavior has previously been documented in the literature, observed during discharge analysis of CNT field emission cathodes [201]. This phenomenon could be attributed to the rising ionization

of residual gas molecules caused by the increasing emission of the dominant tip. As a result, these ions may stimulate nearby tips to emit by raising the local potential through space charge. Additionally, higher rates of desorption resulting from ion bombardment or local temperature increases near the dominant tip because of Joule heating may also serve as explanations. This effect spreads radially, and emission from neighboring tips fluctuates considerably. However, they rarely contribute more than a few thousandths to the integral emission current, while the dominant tip carries up to 6%. Clustering was mainly observed in n^{++} - and n -doped emitters, while it occurred less frequently in p -type FEAs due to the uniformity induced by saturation, which prevents individual tips from dominating.

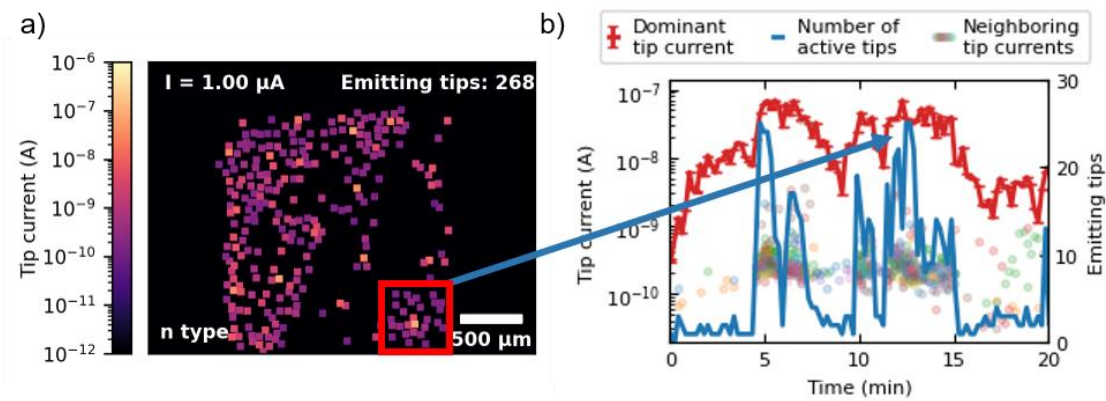


Figure 7.21 Clustering phenomenon illustrated for the n -type FEA during the CCM. In (a), the emission heat map of minute 13 of the CCM is shown. The red box marks the area where the clustering is observed. The graph in (b) plots the current of the dominant tip (red) over the first 20 min of CCM together with the number of emitting tips (blue) inside of the box. The emission currents of the less dominant tips in the box are indicated as multicolored dots. Adapted and reprinted from [103]. Licensed under a Creative Commons Attribution 4.0 (International License: <https://creativecommons.org/licenses/by-nc-nd/4.0/>).

7.4.2 Current distribution during the measurement

Figure 7.22 shows the results of the emission distribution analysis outlined in Chapter 7.3.2 of the three studied emitters. Figure 7.22(a–c) illustrates the evolution of the normalized tip current distribution. Figure 7.22(d–f) displays the number of tips emitting simultaneously at each data point along with the extraction voltage necessary to attain the control current. This number is graphed for each sweep in a different color during the characteristics before and after the CCM. Figure 7.22(g–i) depict the percentage of each bin's contribution to the total integral current at each measurement point. To accomplish this, the cumulative current of all individual tips within each bin was divided by the total current per distribution. Furthermore, the average tip current of the entire FEA is represented in black.

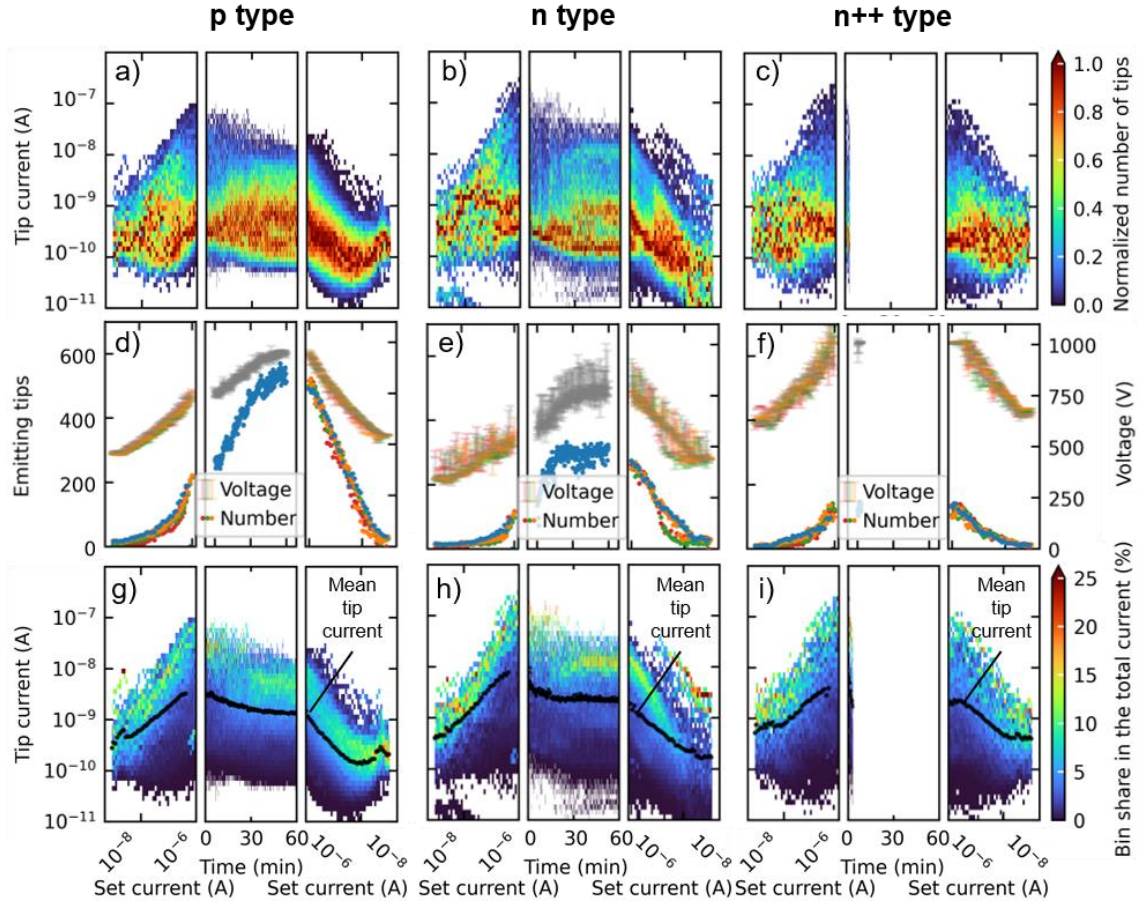


Figure 7.22 (a-c) illustrates the normalized current distribution of the three measured FEAs for every measurement point before, during, and after the CCM as a pseudocolor plot. In (d-f), the number of actively emitting tips is plotted together with the respective extraction voltage. (g-i) represents the center of the distribution in terms of each group's share of the FEA emission current, displaying the accumulated percentage of all the tips in each bin to the integral current. Adapted and reprinted from [103]. Licensed under a Creative Commons Attribution 4.0 (International License: <https://creativecommons.org/licenses/by-nc-nd/4.0/>).

The overall shape of the current distributions in Figure 7.22(a-c) is comparable among all three dopants. During the initial characteristics, the distributions show a high degree of scattering, but stabilize during the CCM. In the subsequent characteristics, the distributions are narrower with less prominent maximum currents. Part of this stabilization is attributable to various conditioning effects [70], [83] affecting the emission of the individual tips during the initial operation. Additionally, from Figure 7.22(d-f), it is apparent that the number of emitting tips increases significantly over the course of the measurement, due to the increasing voltage. Analyzing only the sweeps before the CCM, we observe a rapid and reproducible activation of up to 250 tips for the p-doped FEA with comparable extraction voltages for each sweep. In contrast, for the n^{++} and n FEA, only a limited number of tips contribute to the emission current simultaneously across a wide set current and extraction voltage range, reaching a maximum of about 125 tips. Furthermore, the voltage data points in Figure 7.22(e,f)

regarding n^{++} - and n-type are more widely scattered, indicating more fluctuation and degradation effects between the individual sweeps of the characteristic.

Notable differences in the number of simultaneously emitting tips during the CCM are observed in Figure 7.22(d,e). The p-type FEA continually activates more tips, while the n-type FEA reaches its maximum at approximately 300 individual emitters after around 20 min. At about the same time, distinct levels of strongly represented emitter groups start to form in the distribution course shown in Figure 7.22(b). From this point on, further increase of the extraction voltage induces many of the already active tips to emit higher currents and/or counters their degradation in terms of emission current. As a result, an increase in the number of emitters producing more than 10 nA can be observed in the CCM part of Figure 7.22b, in addition to the numerous tips emitting between 0.1 and 1 nA. Unlike this, the number of tips emitting simultaneously in the p-type FEA increases continuously and reaches over 500 by the end of the CCM. This corresponds to over 55% of the tips in the array. According to Figure 7.22(a), the increased number of tips results in a more continuous distribution of emitted currents. However, it is worth noting that the tips emitting currents within the range of 0.1 to 1 nA are most strongly represented here as well.

Another significant difference between the dopant types can be observed in the maximum currents in Figure 7.22(a–c), reaching values of over 200 nA per tip for the n- and n^{++} -type FEA and only up to 100 nA for the p-type tips during the initial sweeps. However, it cannot be excluded that this difference is due to a possible voltage drop over the space charge region of p-doped emitters in saturation. During CCM, the maximum currents decrease significantly for both the n- and p-type FEA. Note that the n^{++} -type FEA is excluded from the comparison because its CCM ended after two minutes due to the extraction voltage limit. Apart from the expected high degradation of the strongly emitting tips regardless of doping, both FEAs depicted in Figure 7.22(a,b) may display similar behavior due to the potential combination of adsorbates and thermal runaway. The measurements took place at a high chamber pressure of 10^{-5} mbar, which may have caused the accumulation of numerous adsorbates on the surface of the emitting tips during the initial sweeps. The presence of adsorbates away from the tip apex affects the generation of charge carriers on the surface, leading to a possible increase in the current limit of p-type FEAs [190]. Moreover, the presence of adsorbates at the tip apex can lead to a decrease in current due to a change in the work function [83]. As the constant current heats the tip during the initial minutes of CCM, the desorption rate rises. At the same time, the elevated temperature results in a rising generation of charge carriers. Depending on the surface conditions, temperature, and doping, the typical current saturation for p-type FEAs, which is in the range of a few nanoamperes, can be restored (refer

to Figure 7.20, p-type, orange graph), or the tip current continues to increase until it degrades because of thermal runaway (refer to Figure 7.20, p-type, blue graph). For the n-type FEA, various types of degradation can be observed, as demonstrated by its individual tip characteristics presented in Figure 7.20. A more comprehensive analysis of these degradation types will be discussed in Section 7.4.3.

The figures displayed in Figure 7.22(g–i) demonstrate the respective contributions made by each group to the total FEA current. As mentioned before, it is well known in the literature that most of the FEA current is typically carried by a small number of very sharp tips, especially at low voltages [143], [228], [276]. Particularly during the characteristics before the CCM, this is true for all the examined FEAs, independent of the doping. During CCM, however, differences between the p- and n-type emerge. Especially at the beginning, the n-type FEA relies mainly on its tips emitting around 100 nA. However, as the measurement continues and the extraction voltage increases, many of those tips degrade due to their high current load. After approximately 20 min of CCM, a state of equilibrium is reached in which the distributions are centered around the tips emitting slightly more than 10 nA. This phenomenon is less evident for the p-type FEA, as current saturation suppresses excessive emissions from the beginning, forcing the emitter to spread the current load over more tips. This helps to prevent premature burn-out and associated degradation. The equilibrium state, which is achieved after around 30 min, aligns well with the expected current saturation levels typically around 1 nA to 10 nA, as previously stated. However, activating the additional tips requires a higher electric field, causing the p-doped emitter to exhibit a faster increase in extraction voltage than the n-doped one.

Upon closer analysis of the data, it is evident that at the start of the CCM, only 20% of the current for p-type FEAs is carried by tips emitting 10 nA or less. However, as the more highly emitting tips degrade over time, this percentage steadily increases to 90% by minute 30, where it remains until the end of the measurement. Similarly, at the beginning of the CCM, 20% of the set current for the n-type FEA comes from tips emitting 10 nA or less. However, the value increases linearly to only 50% until minute 20 and then fluctuates around this average value. The remaining current share for the n-type emitter of 80% at the beginning of the CCM and 50% at the end is distributed over only 15 and 20 tips, respectively. For the p emitter, however, this value drops from 25 tips carrying 80% initially, to only 5 tips carrying the residual 10% of the integral current by emitting over 10 nA in the end. This study confirms that, firstly, the saturation effects of p-doped emitters can notably enhance the uniformity of FEAs. Secondly, the enhanced uniformity also results in steady emission and prolonged lifespan of FEAs through the spread of current load across numerous tips

7.4.3 Individual Tip IV Characteristics

For a more comprehensive understanding of the degradation of individual tips under constant current load during the one-hour CCM, this section will analyze the IV characteristics of the individual tips before and after CCM in greater detail. The evaluation is based on the methodology outlined in Chapter 7.3.3. The result of the comparative study between the three differently doped emitters is shown in Figure 7.23. In Figure 7.23(a–c) the change in the FEF of a tip as a function of its FEF before CCM is depicted. Tips that did not emit after CCM are shown in red. The data points of tips that were active both times (blue) were fitted linearly, in order to determine their zero crossing. Additionally, their mean change was determined and plotted in brown. Figure 7.23(d–f) illustrates in detail how each of these tips has changed individually, as the FEF after the CCM is plotted against the FEF before. In case of an increase (points above the half line), the respective tip is shown in green, and in case of a deterioration (below the half line) in orange. The mean FEF before and after is plotted in brown. In addition, at $x = 0$, the newly activated tips during the CCM are displayed in blue, and at $y = 0$, those deactivated during the CCM in red.

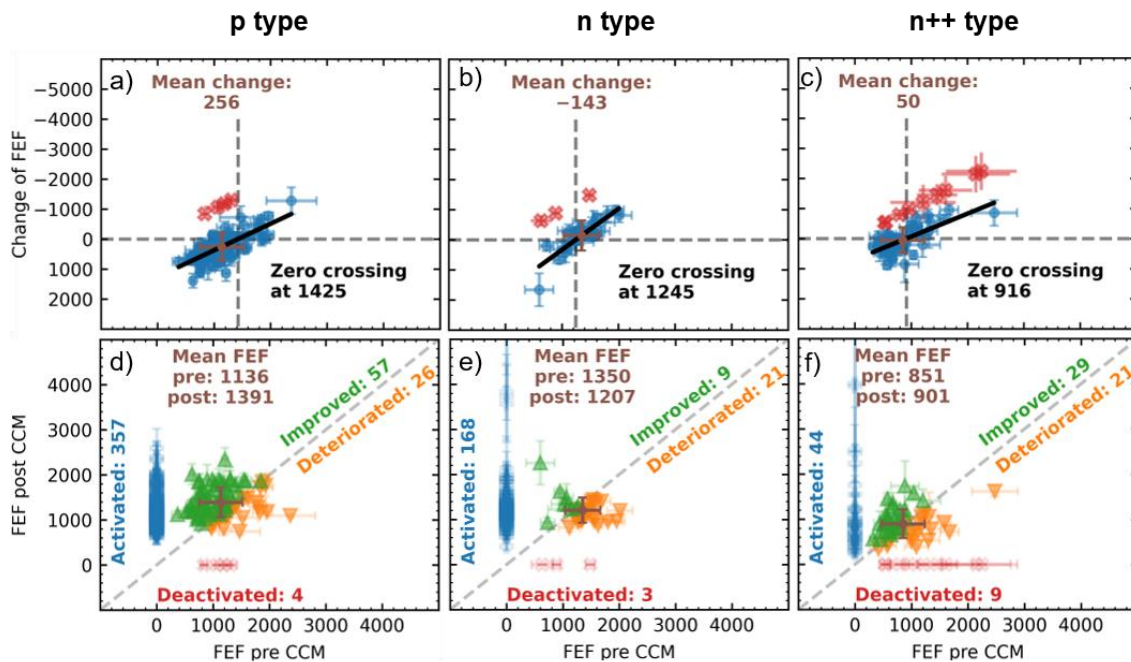


Figure 7.23 Comparative study of the change in single-tip FEFs. In (a–c) the difference between the FEF pre and post CCM is plotted versus the initial FEF. (d–f) show the FEF post CCM versus the FEF pre CCM, together with the newly activated and during the CCM deactivated tips. Note that the CCM of the n⁺⁺-type FEA only lasted for three minutes due to the limited extraction voltage, while the p- and n-type were operated for the full 60 min at 1 μ A. Adapted and reprinted from [103]. Licensed under a Creative Commons Attribution 4.0 (International License: <https://creativecommons.org/licenses/by-nc-nd/4.0/>).

From Figure 7.23(a–c), a clear linear relationship between the initial FEF and degradation (change in FEF) can be observed, independent of doping. Higher FEFs typically correspond to sharper tips and higher electric fields at the apex [51], [53], [96]. Therefore, these emitters tend to emit earlier and more current, resulting as expected in higher degradation. Noteworthy is the zero crossing, which corresponds to the threshold above which a tip is more likely to degrade than to improve. For the n^{++} -type FEA, this threshold FEF is 916, for the n -type it is 1245, and for the p -type it is 1425. This indicates that the current limitation of p -doped emitters might prevent higher FEF tips from degradation. Furthermore, the average change of active tips before and after CCM (plotted in brown) is also positive for the p -type emitter, which indicates an improvement in the average, while the n -type emitter degrades on average. The average change for the n^{++} -type FEA is also slightly positive. However, when interpreting the n^{++} -type data, it is essential to note that this FEA was only subjected to three minutes of CCM. Therefore, the degradation cannot be compared to the other two FEAs, as the total current load is different.

A comparison of Figure 7.23(d–f) reveals that the ratio of improving to degrading tips is significantly higher for the p -doped FEA compared to the two n -doped FEAs. Moreover, the activation of 357 new tips outweighs the omission of only four tips in this case. For the n -type FEA, more of the pre-CCM active emitters degrade than improve. However, only three tips are deactivated while 168 new tips are activated. As for the n^{++} -doped FEA, the degradation to improvement and activation to deactivation ratios are closer to one, indicating significant fluctuations during the two-minute CCM.

A notable observation is made by combining the two plots. It appears that the mean FEFs after CCM of the constantly active tips almost perfectly approach the respective threshold FEFs from the zero crossing of Figure 7.23(a–c) in all three cases. Together with the fact that the number of entirely deactivated tips is almost negligible, it demonstrates that the change of the FEA's emission in current-controlled operation is not only dominated by a gradual deterioration of the sharpest tips in the array. It rather exhibits a burn-in process towards a particular ideal FEF by both improvement and deterioration of the individual tips. Preliminary measurement results obtained at varying integral current levels during the CCM demonstrate a clear dependence of the threshold FEF on the integral current load. However, a detailed study of this phenomenon is beyond the scope of this work, and reference is made to future work.

7.4.4 Conclusion

In this study, three differently doped (p-type with resistivity of 10–20 Ωcm , n-type with resistivity of 10–20 Ωcm and n^{++} -type with resistivity of $<0.005 \Omega\text{cm}$) but morphologically similar silicon FEAs were investigated using a tripartite measurement, consisting of current-controlled current–voltage (IV) characteristics measurement prior and subsequent to a one-hour constant current measurement (CCM) at 1 μA at a chamber pressure of 10^{-5} mbar. The n^{++} -type FEA could only emit the set current for a limited time of about three minutes due to the extraction voltage limit of the setup, which made the outcomes partially incomparable. The analysis of the individual tip emissions revealed a clearly observable saturation behavior of several individual p-type tips, which emitted mostly in the range of 1 to 10 nA. This led to a faster and stronger activation of the array and therefore a more stable and uniform current distribution for this kind of doping. During the measurements, a clustering phenomenon was observed in which highly emitting tips led to an increased activation of the surrounding emitters. This effect is more pronounced for the n- and n^{++} -type FEA, due to their inferior emission uniformity. The analysis of the emission distributions revealed that, in accordance with the literature, the integral emission current is heavily dominated by the strongest emitting tips, especially during the initial characteristics for the n- and n^{++} -type FEA. Continued conditioning during the CCM led to an equilibrium state being reached for both the p-type and n-type. Here, the majority of the total FEA current was carried by a distinct group of tips emitting over 10 nA for the n-type, while for the p-type, the emission centered around slightly lower current levels that were less discrete. A burn-in process was identified as the cause of this equilibrium state by analyzing the individual tip field enhancement factors obtained from the IV characteristics before and after the CCM. It was demonstrated that both the improvement and deterioration of individual tips result in the mean field enhancement factor approaching a current-dependent optimum value.

7.5 Discussion of limits and potential

In this chapter, the newly developed QFEI measurement method was introduced, including its basic idea, metrology, and evaluation methodology. Note, that at this stage of development, the accuracy of the single tip current reconstruction from the integral optical data has not yet been demonstrated. However, the determined magnitudes and the course of the individual emission site currents during the measurement are in good agreement with the literature. Therefore, this measurement is suitable for relative before-and-after comparisons or comparisons between different FEAs on a statistical basis.

Furthermore, these findings only signify an initial step in the development of the QFEI in its basic configuration, as there is significant room for improvement. To enable a broader application, multiple concerns demand attention. First, a triode configuration of the setup may be realized to address the electron energy-dependent sensitivity of the sensor. In this way, the macroscopic energy range can be set to an arbitrary constant level and thus be adapted to the respective requirements. Additionally, this permits the measurement of gated FEAs and expand the range of feasible applications. It could also be used to perform further validation measurements to investigate current and energy dependence of the sensor response in more detail. Another possibility is the integration of electron optics into the triode configuration. Currently, the camera is in close proximity to the FEA, putting the emitter at risk of contamination from material ablated off the sensor's surface. Additionally, thermally induced outgassing near the sensor surface may affect the local pressure and thus the emission properties of the emitters. By integrating an electrostatic einzel lens, the emission of the FEA might be imaged at a greater distance, thus avoiding such influences. First successful measurements in this respect could already be realized by our project partner, but are not part of the work presented here.

The second problem to be addressed concerns the degradation of the camera, as high current densities can cause damage to the sensor surface and even render the camera inoperable. This affects the reliability of optical data used to compute emission site currents, as well as the maximum measurable currents. During the experiments, it was determined that camera degradation had a negligible effect if the single tip currents did not exceed 100 nA for extended periods. For this reason, the maximum emission current of the entire FEA was limited to 1 μ A for all the measurements presented in this work. One solution that may allow higher currents to be measured is to apply a coating to the sensor surface. However, experiments investigating this topic are still ongoing. Preliminary results suggest that a sputtered layer of tungsten with a thickness of 40 nm can significantly enhance the camera's longevity and resistance to high current densities [281]. This method's drawback is that reduced transmission permits the detection of low currents only by considerably increasing the shutter speed and measurement time. To allow for the analysis of particularly small currents, the sensor surface can also be further exposed. *Wilkes et al.* demonstrated that suitable solvents can remove the microlenses and color filter array from the sensors surface [255]. This also facilitates creation of any layer combination of insulator and metallization on the pristine sensor surface to adapt the camera to the specific measurement task depending on the current range to be investigated. Additionally, instead of modifying the Raspberry Pi HQ camera, the measurement

principle outlined in this work can be applied to several other sensors that are commercially available.

Furthermore, there is scope for improvement regarding the acquisition and evaluation of data. It is possible to adjust the measurement speed depending on the desired current measurement range, such as by using a single shutter speed. This would remove the need to adjust the camera's exposure time, resulting in approximately a tenfold reduction in measurement time. Regarding the evaluation of the data, orthodoxy [45] and validity tests [43], [44] can be integrated to automatically check the quality of the measurement. For example, the integration of a recently published online tool [186], [282], [283] would be suitable for this purpose.

8 Conclusions and Outlook

In this thesis, a silicon nanowire (SiNW) based field emitter array (FEA) with an integrated extraction gate was developed. It is intended to be incorporated as a robust and reliable electron source in the high-pressure environment of a handheld X-ray device in future work. The FEA's design was conceived to withstand the required high emission currents ranging from 0.1 to 10 mA at elevated operational pressure levels of 10^{-5} mbar by combining the unique field emission properties of SiNWs with elongated leakage current paths to the gate via insulated trenches between emitter and extractor. Additionally, a novel measurement method was developed, that allows for the quantified detection of individual emission sites' currents during the integral operation of the FEA. This can be used to further optimize the developed devices in future work, e.g., by investigating local current limitation for enhanced emission uniformity.

The literature review, presented in Chapter 3, revealed that many state-of-the-art FEAs are already capable to meet the abovementioned requirements for high-current applications in ultra-high vacuum conditions. However, there is limited data on FEA performance at high operating pressures, which are more commonly encountered in commercial applications. Multiple studies suggest that the conductivity, morphology, and packing density of the emitters have a greater impact on the electron source's performance than the choice of the FEA's substrate material based on the work function. Therefore, silicon was chosen as the base material in this work, due to the well-established manufacturing processes provided by the semiconductor industry, which allow for precise and reproducible formation of a wide variety of emitter morphologies on a wafer scale base.

In Chapter 5, the capabilities of two fundamentally different emitter shapes and corresponding fabrication processes to withstand the given high current carrying capability requirements at elevated pressure levels were investigated in diode configuration measurements. The first process involves orientation-dependent wet-chemical etching of silicon. Arrays of pyramidal tips with nanometric apex radii can be formed by pre-structuring pillars with square bases oriented parallel to the {100} planes of the crystal using a wafer dicer and subsequent etching in tetramethylammonium hydroxide (TMAH). The size of the tips depends on the diameter of the pillars. All measurements presented in this work were obtained from arrays of either 30 x 30 or 60 x 60 tips with an average height of approximately 30 μm . The morphology of the second emitter type, which was newly developed, is based on vertical SiNWs produced through a self-masking process using reactive ion etching and the Bosch

process. In contrast to common practice in the literature, the manufacturing process presented here is self-masking and not based on highly sophisticated lithography. In the first step, photolithography and the Bosch process are used to create mesas with vertical sidewalls on silicon wafers. In a subsequent second Bosch process, the mesas are left unmasked, making them susceptible to combined semi-anisotropic etch removal by physical ablation and chemical erosion. The vertical sidewalls of the mesas reduce the probability of ion impact and thus complete physical removal of the passivation. Consequently, small passivation residues remain on the edges of the mesa, serving as a mask for the formation of the vertical nanowires.

The electrical properties of TMAH-etched and multiple variants of SiNW FEAs were compared using a current dependent lifetime test (CDLT) at a pressure level of 10^{-5} mbar. In this test, three samples of each FEA type are operated for one hour each at increasing current levels. Before and after each constant current operation, a total of seven current controlled current-voltage sweeps are performed, respectively. The statistical evaluation of the test results revealed significant performance variation even within samples of the same type, particularly for the SiNW FEAs. These variations could be attributed to morphological differences between the individual samples resulting from the self-masking fabrication process. Furthermore, the design of the sample holder caused heating of the system, including the measured FEA, due to the power dissipation into the extractor by the electron beam at high emission currents. The temperature increase in the measured FEAs could potentially change the results of this measurement when compared with measurements in a triode configuration, where the anode is thermally decoupled from the emitter. Nevertheless, the measurements demonstrated that some of the fabricated SiNW FEAs are capable of emitting emission currents above the minimum required 100 μA for at least several hours. In contrast, the TMAH-etched FEAs were not able to exceed a maximum emission current of 100 μA at similar electric field strengths. The maximum emission current achieved by the SiNW FEAs, within the maximum available extraction voltage of 1.4 kV, was 2.5 mA. However, it should be noted that, due to the methodology of the test, the corresponding emitters had already completed 20 one-hour constant current operations at lower currents at this time.

Subsequently, the impact of various dopants and doping densities on the emission properties of SiNW FEAs was investigated, in order to identify potential for future device optimization. The measurements based on the CDLT methodology revealed, that lowly n-doped samples reached the highest emission current on average, followed by highly n^{++} -doped and lowly p-doped emitters. Highly p^{++} -doped FEAs exhibited the worst overall performance. These differences could be attributed to the different substrate conductivities, which affect the local emission current limitation in the nanowires. In general, proper current limitation

can lead to an improved emission uniformity in the array and thus potentially increase the lifetime of the FEAs. In the case of the p-type samples, a very clear and reproducible saturation behavior of the integral emission current could be observed during current-voltage measurements when they exceeded a few microamps. This behavior led to a significant increase in the extraction fields required to obtain high emission currents, which ultimately resulted in an inferior CDLT performance of the p-type samples compared to the n-doped FEAs. Nevertheless, this also suggests, that p-type doping may be a viable tool to compensate for morphological differences in the nanowires by effectively limiting the emission current supply of each individual emission site. Therefore, the overall lifetime of p-type FEAs can be superior in current regimes not exceeding the integral saturation current. Furthermore, it was argued, that the p-type dopant concentration of the substrate could be used to adjust the individual emission sites' and thus also the integral current saturation level. However, further investigations will be needed in this regard.

In Chapter 6, a novel FEA design was conceived that integrates an extraction gate around the SiNW emitters. Typically, the surface of the insulating layer between the gate and emitter is a crucial weak point, particularly when operating gated FEAs at high pressures. The accumulation of adsorbates can cause dielectric surface breakdown, flashovers, and/or excessive leakage currents into the gate. To prevent this, the presented design intentionally increases the leakage path by structuring thermally oxidized trenches around the emitters. In this proof of concept, thermally evaporated aluminum was chosen as the gate material since no suitable alternatives were available at the time of this work. A mask set consisting of four photolithography masks was designed based on the planned process sequence. The individual process steps were then introduced and optimized successively. The functionality of the FEAs was found to be particularly dependent on the vertical positioning of the nanowire apices. A two-dimensional electrostatic simulation using COMSOL Multiphysics® demonstrated that electric fields applied to the apices are maximized when they are approximately on the same level or slightly above the extraction gate. After implementing this necessary process optimization, the fabricated emitters were electrically characterized.

During electrical characterization many emitters were found to exhibit short-circuits immediately after installation in the measurement setup, even though no short-circuit could be detected during initial testing with a wafer prober shortly after fabrication. This was attributed to sample handling challenges during transport and installation into the setup, since the elevated emitter apices make the nanowires prone to mechanical damages and failure caused by contamination with conductive particles. Furthermore, many of the initially functional emitters failed randomly after short periods of operation, which could be optimized by

a different choice of gate material. Scanning electron microscopy (SEM) investigation of the samples after measurement indicated that the low melting point of aluminum might promote outgassing of the gate material, which in turn leads to local pressure increase and triggers arcing from the emitter to the gate. The spattering aluminum then carries the risk of short-circuiting the entire FEA if it is unfavorably redeposited on the surface of an insulating trench. Despite these challenges, several samples survived long enough to reveal the potential of the developed FEA, since the gate material induced failure is of random nature. First, a study on the current dependent transmission of the gated SiNW FEAs was conducted based on the CDLT methodology. The maximum emission current was limited to 10 μA during this measurement to protect the picoammeter installed for measuring the intercepted current by the gate from possible flashover-induced damage. The results showed a stable transmission of over 95% during constant current operation for all current levels assessed. The maximum current carrying capabilities of the gated SiNW FEAs were investigated after removing the picoammeter from the setup. During the CDLT, a maximum emission current of 630 μA was achieved. This corresponds to a total operation time of the FEA of about 24 hours at various set current levels and a pressure of 10^{-5} mbar. Five one-hour constant current measurements were conducted at or above 100 μA at this point, without any notable voltage increase during each measurement. This suggests low degradation and high lifetime of the FEA for these current levels. The emission current of 630 μA was achieved at an average extraction voltage of approximately 280 V. It is worth noting that the FEA remained functional after this measurement, as the CDLT had to be manually terminated due to a measurement setup malfunction. A repetition of the measurement was not carried out here, as the problems concerning the sample yield and the measurement setup defect must be resolved first. Nevertheless, these measurements demonstrate, that the gated SiNW FEAs can deliver the minimum required emission current of 100 μA at the desired pressure level of 10^{-5} mbar for at least a few hours. Thus, they are suitable for initial tests in a prototype of a field emission-based portable X-ray device. However, before commercial use, some optimizations still need to be carried out, which will be outlined in the next section.

A decisive factor in future optimization of the gated SiNW FEAs might be the newly developed quantitative field emission imaging (QFEI) measurement method, which was introduced in Chapter 7. The method uses a low-cost Raspberry Pi HQ camera to detect the emission currents of individual tips of an FEA in a spatially resolved and quantifiable manner during the integral operation of the emitter. The measurement signal is likely due to X-ray photons generated by bremsstrahlung from the decelerating electrons upon impact on the sen-

sensor surface. A camera control software was developed, which can efficiently acquire and process raw images captured by the camera. This was used in an SEM test measurement to demonstrate a linear correlation between incident electron beam currents on the sensor and brightness detected by the camera for multiple shutter speeds. A standard measurement setup and procedure was established. During the measurement, images are captured at multiple exposure times (typically 1 ms, 3.15 ms, 10 ms, and 31.5 ms) in order to extend the dynamic range of the camera and allow for detection of electron emission ranging over multiple magnitudes of current. The time required for image acquisition was reduced to a minimum of 4–6 seconds per electrical data point after software optimization. During post-processing, a spot detection algorithm is used to identify each individual emission site and check for pixel saturation. If at least one pixel in a spot is saturated, the entire spot is replaced with the corresponding spot from an image acquired at a lower exposure time without saturated pixels by linearly extrapolating its brightness to the higher exposure time. This way, a composite image with an arbitrarily expandable dynamic range is generated. The extrapolated image can then be used to reconstruct the individual tip currents from the respective spot brightnesses by multiplying them with the quotient of integral current to integral brightness.

A first QFEI measurement was conducted on a lowly n-doped TMAH FEA at a base pressure of 10^{-5} mbar. The measurement consisted of seven current-controlled current-voltage sweeps before and after a one-hour constant current measurement at 1 μ A. Several methods for analyzing this novel type of data were presented and critically discussed. The results were mostly consistent with the predictions from literature, which validates the measurement method. Subsequently, the QFEI was used to study the doping-dependent emission behavior of TMAH FEAs. Three morphologically similar silicon FEAs with different dopings (p-type with resistivity of 10–20 Ω cm, n-type with resistivity of 10–20 Ω cm and n^{++} -type with resistivity of < 0.005 Ω cm) were measured at a base pressure of 10^{-5} mbar following the same tripartite measurement procedure as before. The p-type sample exhibited clear saturation behavior of individual tip characteristics, primarily in the 1 to 10 nA range of emission current. This saturation was found to be attributable for the superior activation of emitting tips, resulting in a more uniform emission current distribution across the array for p-type FEAs. Furthermore, a burn-in phenomenon could be identified by analyzing the field enhancement factors (FEFs) obtained from the individual current-voltage characteristics before and after the constant current measurement. The results indicate that the constant current operation of FEAs does not simply result in the gradual degradation of the highest FEFs of the array. Instead, it trims the individual FEFs of each actively emitting tip towards a common optimum.

During the process, both sharp tips become blunted while blunt tips become sharpened. Preliminary results suggest that the optimal level depends on the emission current and FEA doping.

In conclusion, this work has established the foundations for the development of a field emission-based electron source for high-current applications under high pressure environments. However, to enable gated SiNW FEAs to compete with commercial products based on thermal emission cathodes, the manufacturing process yield and emitter lifetime must be improved. The choice of gate material is likely the most influential factor and also the easiest to change. To reduce the random failures of the FEAs after short operating times, it is recommended to use a material with a higher melting point that is less prone to outgassing. Furthermore, optimizing the sample transport and the design of the sample holder in the measurement setup can prevent mechanical damage to the samples and contamination of the sample surface. An additional insulating layer, such as mechanically robust silicon nitride, could be implemented on the FEA surface. This would reduce the probability of a short circuit caused by conductive connections between the emitter and gate, such as conductive particles or broken nanowires. After implementing these optimizations, it is necessary to conduct further measurements to characterize the electrical properties of the FEAs. The primary measurement should determine the actual lifetime of the FEAs through constant current operation in the targeted current range of 0.1 to 10 mA until complete device failure. Additionally, a measurement under ultra-high vacuum conditions would be useful to compare the developed FEAs with the state of the art.

The second major task concerns the QFEI method. In its current state, the method cannot yet be used for optimization of the gated SiNW FEAs. This is primarily due to the sensor's inability to withstand high local current densities. Initial tests have already demonstrated that a thin metal coating on the sensor surface can address this issue. In combination with a preceding complete exposure of the pristine sensor surface, a layer sequence of insulator and metallization could also prove beneficial here. However, the exact dimensions of the layers and suitable materials to achieve the highest possible signal transmission with sufficient current density resilience remains to be determined. Furthermore, a circuit must be developed that enables current-controlled measurement of gated FEAs in triode configuration with the camera. These optimizations will allow for unique measurements to be performed on the gated SiNW FEAs. First, it would be particularly interesting to investigate the effectiveness of local current limitations in the FEA. Both the possible morphologically induced limitation by the nanowires, as well as a dopant-induced limitation could be optimized to the desired

working range of the electron source using the unprecedented insights of this measurement method. Apart from this, the superposition of the FEA characteristic curve consisting of differently performing individual emitters with various FEFs could also be investigated. As a result, the newly developed method could not only be used in the future to make field emission-based electron sources commercially viable, but could also contribute to the general understanding of FEAs by providing unprecedented insights into their unique emission behavior.

Appendix

A. Process Sheets for Field Emitter Array Fabrication

A.1 TMAH FEA

Table 2: Process sheet for TMAH FEAs.

Nr.	Process step (Tool)	Purpose	Parameters	
1	Wafer dicing (Wafer dicer)	Cut wafer into 4 cm x 4 cm samples	Orientation:	45° / 135° (to flat)
			Pitch:	40 000 µm
			Blade height:	70 µm
2	Wafer dicing (Wafer dicer)	Pre-structure pillars	Orientation:	0° / 90° (to edge)
			Pitch:	66,7 µm
			Blade height:	570 µm
3	RCA1 (Wet bench)	Cleaning 1	Medium:	H ₂ O : NH ₃ (25%) : H ₂ O ₂ (30%)
			Ratio:	5 : 1 : 1
			Temperature:	80 °C (hot plate)
			Time:	10 min
4	RCA2 (Wet bench)	Cleaning 2	Medium:	H ₂ O : HCl (37%) : H ₂ O ₂ (30%)
			Ratio:	5 : 1 : 1
			Temperature:	80 °C (hot plate)
			Time:	10 min
5	HF-Dip (Wet bench)	Remove oxide	Medium:	HF (1%)
			Temperature:	RT
			Time:	1 min
6	TMAH etch (Wet bench)	Structure tips	Medium:	TMAH (25%) : H ₂ O
			Ratio:	1 : 4 (→ 20% TMAH)
			Temperature:	70 °C (74 °C water bath)
			Time:	25–32 min (on sight)

A.2 Silicon nanowire FEA

Table 3: Process sheet for silicon nanowire FEAs.

Nr.	Process step (Tool)	Purpose	Parameters					
1		Mesas						
1.1	Lithography (Mask aligner)	Resist for mesa structures						
A	Coat (Spin coater)		Pretreatment:	HMDS 5 min				
			Resist:	AZ 10 XT (Microchemicals)				
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rpm, 60 s				
			Prebake:	110 °C, 3 min				
			Rehydration:	> 30 min				
B	Expose (Mask aligner)		Mask:	SiNW – SiNW				
			Contact:	hard contact				
			Time:	27.5 s				
C	Develop (Wet bench)		Developer:	AZ 326 MIF (Microchemicals)				
			Temperature:	RT				
			Time:	10 min				
1.2	RIE (Plasmalab 100)	Bosch process etch of mesa structures	Recipe:	RIE/RIE				
			Cycles: 150					
			Step	Time (s)	Q (sccm) C ₄ F ₈ /SF ₆	CCP (W)	ICP (W)	Valve Pos.
			0	5	50/20	45	300	20
			Dep	5	100/1	10	300	48
			Etch	2	0/80	30	500	43.2

1.3	Strip resist	Strip resist	Medium 1:	Aceton				
	(Wet bench)		Temperature:	RT				
			Time:	10 min (with US)				
			Medium 2:	IPA				
			Temperature:	RT				
			Time:	10 min (with US)				
2		Nanowires						
2.1	Lithography	Resist for die edge protection during nanowire etch						
	(Mask aligner)							
	a	Coat	Pretreatment:	HMDS 5 min				
		(Spin coater)	Resist:	AZ 10 XT (Microchemicals)				
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rmp, 60 s				
			Prebake:	110 °C, 3 min				
			Rehydration:	> 30 min				
	b	Expose	Mask:	SiNW – Oxide				
		(Mask aligner)	Contact:	hard contact				
			Time:	180 s				
	c	Develop	Developer:	AZ 326 MIF (Microchemicals)				
		(Wet bench)	Temperature:	RT				
			Time:	15 min				
2.2	RIE	Bosch process etch for nanowires	Recipe:	RIE/RIE				
	(Plasmalab 100)		Cycles:200					
			Step	Time (s)	Q (sccm) C4F8/SF6	CCP (W)	ICP (W)	Valve Pos.
			0	5	50/20	45	300	20
			Dep	5	100/1	10	300	48
			Etch	2	0/80	30	500	43.2

2.3	Strip resist	Strip resist	Medium 1:	Aceton				
	(Wet bench)		Temperature:	RT				
			Time:	10 min (with US)				
			Medium 2:	IPA				
			Temperature:	RT				
			Time:	10 min (with US)				
2.4	RIE	Depassivation	Recipe:	Veraschen Andi				
	(Plasmalab 80)		Step	Time (s)	Q (sccm) O ₂	CCP (W)	ICP (W)	Pressure (mtorr)
			1	600	30	10	300	50

A.3 Gated silicon nanowire FEA

Table 4: Process sheet for gated silicon nanowire FEAs.

Nr.	Process step (Tool)	Purpose	Parameters					
1	Mesas							
1.1	Lithography (Mask aligner)	Resist for mesa structures						
a	Coat (Spin coater)		Pretreatment:	HMDS 5 min				
			Resist:	AZ 10 XT (Microchemicals)				
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rmp, 60 s				
			Prebake:	110 °C, 3 min				
			Rehydration:	> 30 min				
b	Expose (Mask aligner)		Mask:	SiNW – SiNW				
			Contact:	vacuum contact				
			Time:	17.5 s				
c	Develop (Wet bench)		Developer:	AZ 326 MIF (Microchemicals)				
			Temperature:	RT				
			Time:	9 – 10 min				
1.2	RIE (Plasmalab 100)	Bosch process etch of mesa structures	Recipe:	RIE/RIE				
			Cycles:60					
			Step	Time (s)	Q (sccm) C ₄ F ₈ /SF ₆	CCP (W)	ICP (W)	Valve Pos.
			0	5	50/20	45	300	20
			Dep	5	100/1	10	300	48
			Etch	2	0/80	30	500	43.2

1.3	Strip resist	Strip resist	Medium 1:	Aceton				
	(Wet bench)		Temperature:	RT				
			Time:	10 min (with US)				
			Medium 2:	IPA				
			Temperature:	RT				
			Time:	10 min (with US)				
1.4	RIE	Remove residual passivation from Bosch process	Recipe:		Veraschen Andi			
	(Plasmalab 80)		Step	Time (s)	Q (sccm) O ₂	CCP (W)	ICP (W)	Pressure (mTorr)
			1	600	30	10	0	20
2		Trenches						
2.1	Lithography	Resist for trenches						
	(Mask aligner)							
a	Coat		Pretreatment:		HMDS 5 min			
	(Spin coater)		Resist:		AZ 10 XT (Microchemicals)			
			Spin coating:		(1) 500 rpm, 8 s (2) 1800 rmp, 60 s			
		Prebake:		110 °C, 3 min				
		Rehydration:		> 30 min				
b	Expose	Mask:	SiNW – Trenches					
	(Mask aligner)	Contact:	vacuum contact					
		Time:	17.5 s					
c	Develop	Developer:	AZ 326 MIF (Microchemicals)					
	(Wet bench)	Temperature:	RT					
		Time:	9–10 min					

2.2	RIE	Bosch process etch of trenches	Recipe:	RIE/RIE				
	(Plasmalab 100)		Cycles: 150					
			Step	Time (s)	Q (sccm) C ₄ F ₈ /SF ₆	CCP (W)	ICP (W)	Valve Pos.
			0	5	50/20	45	300	20
			Dep	5	100/1	10	300	48
			Etch	2	0/80	30	500	43.2
2.3	Strip resist	Strip resist	Medium 1:	Aceton				
	(Wet bench)		Temperature:	RT				
			Time:	10 min (with US)				
			Medium 2:	IPA				
			Temperature:	RT				
			Time:	10 min (with US)				
3		Dicing mark-ers						
3.1	Lithography	Resist for dic-ing markers on the backside						
	(Mask aligner)							
a	Coat		Pretreatment:	HMDS 5 min				
	(Spin coater)		Resist:	AZ 10 XT (Microchemicals)				
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rmp, 60 s				
			Prebake:	110 °C, 3 min				
			Rehydration:	> 30 min				
b	Expose		Mask:	SiNW – Oxide				
	(Mask aligner)		Contact:	soft contact				
			Time:	25 s				

c	Develop		Developer:	AZ 326 MIF (Microchemicals)					
	(Wet bench)		Temperature:	RT					
			Time:	9 – 10 min					
3.2	RIE (Plasmalab 100)	Bosch process etch for dicing markers on backside	Recipe:	RIE/RIE					
			Cycles:	200					
			Step	Time (s)	Q (sccm) C ₄ F ₈ /SF ₆	CCP (W)	ICP (W)	Valve Pos.	
			0	5	50/20	45	300	20	
			Dep	5	100/1	10	300	48	
			Etch	2	0/80	30	500	43.2	
3.3	Strip resist (Wet bench)	Strip resist	Medium 1:	Aceton					
			Temperature:	RT					
			Time:	10 min (with US)					
			Medium 2:	IPA					
			Temperature:	RT					
			Time:	10 min (with US)					
4		Insulator							
4.1	RCA clean (Wet bench)	Cleaning for oxidation	Medium1:	H ₂ O : NH ₃ (25%) : H ₂ O ₂ (30%)					
			Ratio:	5 : 1 : 1					
			Temperature:	80 °C (Hotplate)					
			Time:	10 min					
			Medium 2:	H ₂ O : HCl(36%) : H ₂ O ₂ (30%)					
			Ratio:	5 : 1 : 1					
			Temperature:	80 °C (Hotplate)					
			Time:	10 min					

4.2	Thermal oxidation (Oxidation furnace)	Insulator	Recipe: OX105030.csv2.csv Temperature: 1050 °C Time: 240 min Type: wet oxidation O ₂ -flow: 4 slm H ₂ -flow: 7 slm Target thickness: ~ 700 – 900 nm
4.3	Lithography (Mask aligner)	Resist for oxide structuring	
a	Coat (Spin coater)		Pretreatment: HMDS 5 min Resist: AZ 10 XT (Microchemicals) Spin coating: (1) 500 rpm, 8 s (2) 1800 rpm, 60 s Prebake: 110 °C, 3 min Rehydration: > 30 min
b	Expose (Mask aligner)		Mask: SiNW – Oxide Contact: soft contact Time: 27.5 s
c	Develop (Wet bench)		Developer: AZ 326 MIF (Microchemicals) Temperature: RT Time: 9–10 min
4.4	Oxide structuring (Wet bench)	Insulator structuring	Medium: Ammonium Fluoride Etching Mixture (Honeywell) Temperature: RT Time: 10–15 min (with US)

4.5	Strip resist (Wet bench)	Strip resist	Medium 1:	Aceton
			Temperature:	RT
			Time:	10 min (with US)
			Medium 2:	IPA
			Temperature:	RT
			Time:	10 min (with US)
5	Gate			
5.1	Aluminum Evaporation (Aluminum evaporation tool)	Gate deposition	Recipe: WSV-Controller	500 nm
			Type:	Resistance evaporation
5.2	Lithography (Mask aligner)	Resist for Gate structuring		
a	Coat (Spin coater)		Pretreatment:	HMDS 5 min
			Resist:	AZ 10 XT (Microchemicals)
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rpm, 60 s
			Prebake:	110 °C, 3 min
			Rehydration:	> 30 min
b	Expose (Mask aligner)		Mask:	SiNW – Gate
			Contact:	soft contact
			Time:	27.5 s
c	Develop (Wet bench)		Developer:	AZ 326 MIF (Microchemicals)
			Temperature:	RT
			Time:	9–10 min
5.3	Aluminum structuring (Wet bench)	Gate Structuring	Medium:	Phosphoric Acid Etching Mixture (Honeywell)
			Temperature:	45 °C (Waterbath)
			Time:	~ 1 min (with US)

5.4	Strip resist	Strip resist	Medium 1:	Aceton					
	(Wet bench)		Temperature:	RT					
			Time:	10 min (with US)					
			Medium 2:	IPA					
			Temperature:	RT					
			Time:	10 min (with US)					
6	Nanowires								
6.1	Lithography	Resist for nan-							
	(Mask aligner)	owire for-							
		mation							
a	Coat		Pretreatment:	HMDS 5 min					
	(Spin coater)		Resist:	AZ 10 XT (Microchemicals)					
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rmp, 60 s					
			Prebake:	110 °C, 3 min					
			Rehydration:	> 30 min					
b	Expose		Mask:	SiNW – Oxide					
	(Mask aligner)		Contact:	soft contact					
			Time:	27.5 s					
c	Develop		Developer:	AZ 326 MIF (Microchemicals)					
	(Wet bench)		Temperature:	RT					
			Time:	9–10 min					
6.2	RIE	Bosch process	Recipe:	RIE/RIE					
	(Plasmalab 100)	etch for nan-	Cycles: 100						
		owires		Step	Time (s)	Q (sccm) C ₄ F ₈ /SF ₆	CCP (W)	ICP (W)	Valve Pos.
				0	5	50/20	45	300	20
				Dep	5	100/1	10	300	48
				Etch	2	0/80	30	500	43.2

6.3	Strip resist	Strip resist	Medium 1:	Aceton				
	(Wet bench)		Temperature:	RT				
			Time:	10 min (with US)				
			Medium 2:	IPA				
			Temperature:	RT				
			Time:	10 min (with US)				
6.4	RIE	Depassivation	Recipe:	Veraschen Andi				
	(Plasmalab 80)		Step	Time (s)	Q (sccm) O ₂	CCP (W)	ICP (W)	Pressure (mtorr)
			1	600	30	10	300	50

A.4 Gated TMAH FEA

Table 5: Process sheet for gated TMAH FEAs.

Nr.	Process step (Tool)	Purpose	Parameters					
1		Mesas						
1.1	Lithography (Mask aligner)	Resist for mesa structures						
a	Coat (Spin coater)		Pretreatment:	HMDS 5 min				
			Resist:	AZ 10 XT (Microchemicals)				
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rpm, 60 s				
			Prebake:	110 °C, 3 min				
			Rehydration:	> 30 min				
b	Expose (Mask aligner)		Mask:	SiNW – SiNW				
			Contact:	vacuum contact				
			Time:	17.5 s				
c	Develop (Wet bench)		Developer:	AZ 326 MIF (Microchemicals)				
			Temperature:	RT				
			Time:	9–10 min				
1.2	RIE (Plasmalab 100)	Bosch process etch of mesa structures	Recipe:	RIE/RIE				
			Cycles: 60					
			Step	Time (s)	Q (sccm) C ₄ F ₈ /SF ₆	CCP (W)	ICP (W)	Valve Pos.
			0	5	50/20	45	300	20
			Dep	5	100/1	10	300	48
			Etch	2	0/80	30	500	43.2

1.3	Strip resist	Strip resist	Medium 1:		Aceton			
	(Wet bench)		Temperature:		RT			
			Time:		10 min (with US)			
			Medium 2:		IPA			
			Temperature:		RT			
			Time:		10 min (with US)			
1.4	RIE	Remove residual pas-	Recipe:		Veraschen Andi			
	(Plasmalab 80)	sivation from Bosch pro-	Step	Time (s)	Q (sccm) O ₂	CCP (W)	ICP (W)	Pres- sure (mTorr)
		cess	1	600	30	10	0	20
2		Trenches						
2.1	Lithography (Mask aligner)	Resist for trenches						
a	Coat		Pretreatment:		HMDS 5 min			
	(Spin coater)		Resist:		AZ 10 XT (Microchemicals)			
			Spin coating:		(1) 500 rpm, 8 s (2) 1800 rmp, 60 s			
			Prebake:		110 °C, 3 min			
			Rehydration:		> 30 min			
b	Expose		Mask:		SiNW – Trenches			
	(Mask aligner)		Contact:		vacuum contact			
			Time:		17.5 s			
c	Develop		Developer:		AZ 326 MIF (Microchemicals)			
	(Wet bench)		Temperature:		RT			
			Time:		9–10 min			

2.2	RIE (Plasmalab 100)	Bosch process etch of trenches	Recipe:	RIE/RIE				
			Cycles: 150					
			Step	Time (s)	Q (sccm) C ₄ F ₈ /SF ₆	CCP (W)	ICP (W)	Valve Pos.
			0	5	50/20	45	300	20
			Dep	5	100/1	10	300	48
			Etch	2	0/80	30	500	43.2
2.3	Strip resist (Wet bench)	Strip resist	Medium 1:	Aceton				
			Temperature:	RT				
			Time:	10 min (with US)				
			Medium 2:	IPA				
			Temperature:	RT				
			Time:	10 min (with US)				
3		Dicing markers						
3.1	Lithography (Mask aligner)	Resist for dicing markers on the back-side						
a	Coat (Spin coater)		Pretreatment:	HMDS 5 min				
			Resist:	AZ 10 XT (Microchemicals)				
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rmp, 60 s				
			Prebake:	110 °C, 3 min				
			Rehydration:	> 30 min				
b	Expose (Mask aligner)		Mask:	SiNW – Oxide				
			Contact:	soft contact				
			Time:	25 s				

c	Develop		Developer:	AZ 326 MIF (Microchemicals)				
	(Wet bench)		Temperature:	RT				
			Time:	9–10 min				
3.2	RIE (Plasmalab 100)	Bosch process etch for dicing markers on backside	Recipe:	RIE/RIE				
			Cycles:	200				
			Step	Time (s)	Q (sccm) C ₄ F ₈ /SF ₆	CCP (W)	ICP (W)	Valve Pos.
			0	5	50/20	45	300	20
			Dep	5	100/1	10	300	48
			Etch	2	0/80	30	500	43.2
3.3	Strip resist (Wet bench)	Strip resist	Medium 1:	Aceton				
			Temperature:	RT				
			Time:	10 min (with US)				
			Medium 2:	IPA				
			Temperature:	RT				
			Time:	10 min (with US)				
4		Insulator						
4.1	RCA clean (Wet bench)	Cleaning for oxidation	Medium1:	H ₂ O : NH ₃ (25%) : H ₂ O ₂ (30%)				
			Ratio:	5 : 1 : 1				
			Temperature:	80 °C (Hotplate)				
			Time:	10 min				
			Medium 2:	H ₂ O : HCl(36%) : H ₂ O ₂ (30%)				
			Ratio:	5 : 1 : 1				
			Temperature:	80 °C (Hotplate)				
			Time:	10 min				

4.2	Thermal Oxidation (Oxidation furnace)	Insulator	Recipe: OX105030.csv2.csv Temperature: 1050 °C Time: 240 min Type: wet oxidation O ₂ -flow: 4 slm H ₂ -flow: 7 slm Target thickness: ~ 700–900 nm
4.3	Lithography (Mask aligner)	Resist for oxide structuring	
a	Coat (Spin coater)		Pretreatment: HMDS 5 min Resist: AZ 10 XT (Microchemicals) Spin coating: (1) 500 rpm, 8 s (2) 1800 rpm, 60 s Prebake: 110 °C, 3 min Rehydration: > 30 min
b	Expose (Mask aligner)		Mask: SiNW – Oxide Contact: soft contact Time: 27.5 s
c	Develop (Wet bench)		Developer: AZ 326 MIF (Microchemicals) Temperature: RT Time: 9–10 min
4.4	Oxide structuring (Wet bench)	Insulator structuring	Medium: Ammonium Fluoride Etching Mixture (Honeywell) Temperature: RT Time: 10–15 min (with US)

4.5	Strip resist (Wet bench)	Strip resist	Medium 1:	Aceton
			Temperature:	RT
			Time:	10 min (with US)
			Medium 2:	IPA
			Temperature:	RT
			Time:	10 min (with US)
5	Tip etch			
5.1	RCA clean (Wet bench)	Cleaning for oxidation	Medium1:	H ₂ O : NH ₃ (25%) : H ₂ O ₂ (30%)
			Ratio:	5 : 1 : 1
			Temperature:	80 °C (Hotplate)
			Time:	10 min
			Medium 2:	H ₂ O : HCl(36%) : H ₂ O ₂ (30%)
			Ratio:	5 : 1 : 1
			Temperature:	80 °C (Hotplate)
			Time:	10 min
5.2	HF-Dip (Wet bench)	Remove oxide	Medium:	HF (1%)
			Temperature:	RT
			Time:	1 min
5.3	TMAH etch (Wet bench)	Structure tips	Medium:	TMAH (25%) : H ₂ O
			Ratio:	1 : 4 (→ 20% TMAH)
			Temperature:	70 °C (74 °C water bath)
			Time:	90 s (on sight)
6	Gate			
6.1	Aluminum Evaporation (Aluminum evaporation tool)	Gate depo- sition	Recipe: WSV-Controller	500 nm
			Type:	Resistance evaporation

6.2	Lithography (Mask aligner)	Resist for Gate struc- turing		
a	Coat (Spin coater)		Pretreatment:	HMDS 5 min
			Resist:	AZ 10 XT (Microchemicals)
			Spin coating:	(1) 500 rpm, 8 s (2) 1800 rpm, 60 s
			Prebake:	110 °C, 3 min
			Rehydration:	> 30 min
b	Expose (Mask aligner)		Mask:	SiNW – Gate
			Contact:	soft contact
			Time:	27.5 s
c	Develop (Wet bench)		Developer:	AZ 326 MIF (Microchemicals)
			Temperature:	RT
			Time:	9–10 min
6.3	Aluminum structuring (Wet bench)	Gate Structur- ing	Medium:	Phosphoric Acid Etching Mixture (Honeywell)
			Temperature:	45 °C (Waterbath)
			Time:	~ 1 min (with US)
5.4	Strip resist (Wet bench)	Strip resist	Medium 1:	Aceton
			Temperature:	RT
			Time:	10 min (with US)
			Medium 2:	IPA
			Temperature:	RT
			Time:	10 min (with US)

B. List of Tools for Sample Fabrication and Inspection

- Wafer dicer:
AD2000T – ACCRETECH GmbH
- Spin coater:
PLM7XX – Ramgrabber
- Mask aligner:
MA6 – SÜSS MICROTEC SE
- Reactive ion etcher for silicon:
Plasmalab 100 – Oxford Instruments
- Reactive ion etcher for insulators:
Plasmalab 80 – Oxford Instruments
- Scanning electron microscope 1:
JSM-7610FPlus – JEOL
- Scanning electron microscope 2:
CLARA – Tescan
- Focused ion beam scanning electron microscope:
Crossbeam 550 FIB-SEM – Zeiss
- Aluminum evaporation tool:
VERA 450 – VTD Vakuumtechnik Dresden GmbH

C. Preliminary Results on Gated TMAH Field Emitter Arrays

As mentioned in Chapter 6.1, the set of masks designed in this thesis was intended to work for both gated SiNW FEAs and gated TMAH FEAs. However, the order in which the masks are applied, and the overall process flow must be adjusted for this. A detailed process flow for the fabrication of gated TMAH FEAs, including all parameters, can be found in Table 5 in Appendix A. Figure 8.1 illustrates the key processing steps in a schematic representation.

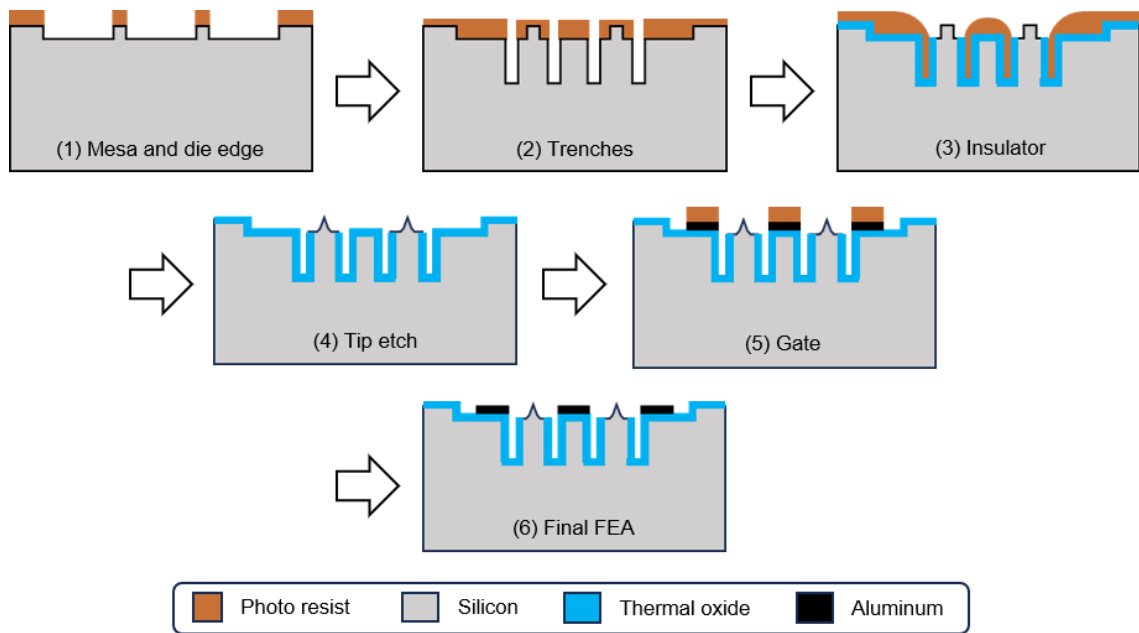


Figure 8.1 Schematic cross-section diagram of the key processing steps for the fabrication of gated TMAH field emitter arrays.

The primary distinction between this process and the ungated TMAH FEAs is that the mesa structure's pre-patterning is accomplished using the Bosch process in this case. This allows for smaller mesa diameters to be achieved and for wafer processing in cleanroom conditions. In comparison to the process flow of the gated SiNW FEAs, the formation of the emitting structure, must be performed before the gate material deposition, since the pyramidal emitters are etched with TMAH, which also etches aluminum and photoresist. Figure 8.2 shows SEM images of the first successful gated TMAH FEA samples.

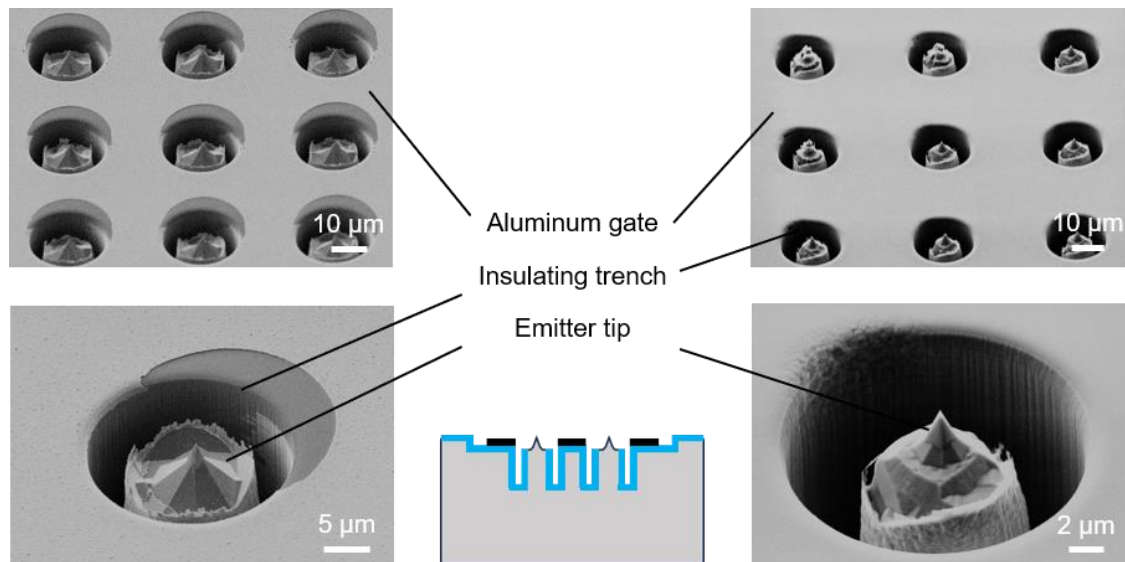


Figure 8.2 SEM images of a CD10G5 (left) and CD5G5 (right) sample obtained with the so far unoptimized process flow. The bottom center depicts the cross-section schematic for both types.

During the first measurements for electrical characterization based on the CDLT, a short circuit occurred at the latest at 1.6 μA in all samples measured. It was also not possible to measure the transmission, which is why leakage currents cannot be ruled out in these measurements. Optimization of these results could be part of future work.

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List of Publications

Peer Reviewed Journals

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