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Carrier Lifetime Measurements in Semiconductors through the Microwave Photoconductivity Decay Method

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Dear Editor
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Please find here a revised manuscript of our paper "Protocol of Carrier Lifetime Measurements in Semiconductors through the Microwave Photoconductivity Decay Method" submitted to Journal of Visualized Experiments.
We hope that this revised version will be satisfied for the editor and the reviewers.

We would be very grateful if you consider it for publication in the journal.

Sincerely,
Masashi Kato

Authors: Takato Asada, Yoshihito Ichikawa, Masashi Kato

Title: Protocol of Carrier Lifetime Measurements in Semiconductors through the Microwave Photoconductivity Decay Method

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TITLE:

Carrier Lifetime Measurements in Semiconductors through the Microwave Photoconductivity Decay Method

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KEYWORDS:

microwave photoconductivity decay method, career lifetime, semiconductor material, time resolved measurement, defect characterization, surface recombination

SUMMARY:

As one of the important physical parameters in semiconductors, carrier lifetime is measured herein via a protocol employing the microwave photoconductivity decay method.

ABSTRACT:

This work presents a protocol employing the microwave photoconductivity decay (μ-PCD) for measurement of the carrier lifetime in semiconductor materials, especially Si. In principle, excess carriers in the semiconductor generated via excitation recombine with time and, subsequently, return to the equilibrium state. The time constant of this recombination is known as the carrier lifetime, an important parameter in semiconductor materials and devices that requires a noncontact and nondestructive measurement ideally achieved by the μ-PCD. During irradiation of a sample, a part of the microwave is reflected by the semiconductor sample. Microwave reflectance depends on the sample conductivity, which is attributed to the carriers. Therefore, the time decay of excess carriers can be observed through detection of the reflected microwave intensity, whose decay curve can be analyzed for estimation of the carrier lifetime. Results confirm the suitability of the μ-PCD protocol in measuring the carrier lifetime in semiconductor materials and devices.

INTRODUCTION:

Excess carriers in semiconductors are optically excited by the injection of photons with energy larger than the gap between the conduction and valence bands. Excited excess carriers, then,

disappear by an electron–hole recombination within a time constant known as the carrier lifetime, which greatly affects the performance of semiconductor devices during operation. As one of the important parameters for semiconductor devices and materials, the carrier lifetime is very sensitive to the presence of defects in these materials, and further requires a convenient method of evaluation. J. Warman and M. Kunst developed a transient technique they named the time resolved microwave conductivity (TRMC), which involves microwave absorption to follow charge carrier dynamics in semiconductors[1]. Other researchers proposed the transient photo conductivity (TPC), otherwise known as the microwave photoconductivity decay (μ-PCD), which is the commonly adopted material qualification technique in semiconductor industries due to its noncontact and nondestructive measurements of the carrier lifetime. In particular, for silicon carbide (SiC), three major techniques are applicable: μ-PCD, time resolved photoluminescence (TR-PL), and time resolved free carrier absorption (TR-FCA)[2–7]. Among these techniques, μ-PCD is the most widely employed because compared to the other two as it exhibits surface roughness insensitivity (i.e., measurable for any given various surface roughness[8–10]) and high signal sensitivity for excited carriers (i.e., using an optimum microwave component). In general, μ-PCD has been preferred for carrier lifetime measurement in SiC and other semiconductor materials[2,5,6,11–19].

The measurement protocol and principle of μ-PCD[1,20,21] is detailed here. In principle, it uses a reflected microwave as a probe. Here, the microwave reflectance of a sample $R(\sigma)$ is equivalent to the ratio between the reflected microwave intensity $P(\sigma)$ and the incident microwave intensity P_{in} as expressed by Equation 1:

$$R(\sigma) = \frac{P(\sigma)}{P_{in}} \quad (1)$$

By irradiation of a pulse laser, the conductivity of a sample σ changes to $\sigma + \Delta\sigma$; likewise, $R(\sigma)$ transforms to $R(\sigma + \Delta\sigma)$. Thus, ΔR is given by Equation 2:

$$\Delta R = R(\sigma + \Delta\sigma) - R(\sigma) = \frac{P(\sigma+\Delta\sigma) - P(\sigma)}{P_{in}} = \frac{\Delta P}{P_{in}} \quad (2)$$

In a perturbation (small $\Delta\sigma$) approximation, $R(\sigma + \Delta\sigma)$ is developed in Taylor series to yield

$$\frac{\Delta P}{P_{in}} = \left[\frac{\delta R(\sigma)}{\delta \sigma}\right] \Delta\sigma, \quad (3)$$

while $\Delta\sigma$ becomes

$$\Delta\sigma = q\left(\mu_p + \mu_n\right)\Delta p, \quad (4)$$

where q is the elementary charge, μ_p is the hole mobility, μ_n is the electron mobility, and Δp is the excess carrier concentration. From the preceding equations, ΔR and Δp are related by

$$\Delta R \propto q\left(\mu_p + \mu_n\right)\Delta p. \quad (5)$$

The dependence of microwave reflectance on excess carrier concentration allows μ-PCD to observe the time decay of excess carriers, which we can use to estimate the carrier lifetime of semiconductor materials.

PROTOCOL:

1. Preparation of the sample

1.1. Prepare an n-type 4H-SiC epilayer (**Table of Materials**).

1.2. Wash the sample with acetone and then with water, each for 5 min, using an ultrasonic washer.

1.3. Use a nitrogen gun to remove moisture on the sample surface.

2. Preparation of aqueous solutions

2.1. Prepare 1 M each of H_2SO_4, HCl, Na_2SO_4, NaOH, or HF at 1 wt% concentration. Select and prepare an aqueous solution to be measured.

2.2. Prepare a quartz cell with 5 mm (length) x 20 mm (width) x 40 mm (height) dimensions and then pour the aqueous solution into it. Put the prepared sample into the cell and then immerse it into the aqueous solution.

NOTE: At least 4 mL of the aqueous solution in the quartz cell is required for the sample to be immersed entirely. When changing the solution, treat the sample with ultrasonic cleaning using acetone and pure water.

3. Preparation of the measuring equipment

3.1. Turn on the power supply of the 266-nm pulsed laser to excite the light source. Afterwards, set the laser mode on standby.

3.2. Connect the pulsed laser and an oscillator through a Bayonet Neill-Concelman (BNC) cable. Turn on the oscillator and input a 100 Hz pulse wave to the pulsed laser.

3.3. Connect a photodiode for trigger acquisition through a trigger input channel of the oscilloscope with a BNC cable.

3.4. Turn on the power supply of the photodiode.

3.5. Irradiate the pulsed laser and place the aperture of the microwave waveguide on the optical path of the laser light at direction normal to the light.

CAUTION: In the latter process, the experimenter should wear safety eye glasses during laser irradiation.

3.6. Install a half-mirror on the optical path of the pulsed laser, as shown in **Figure 1**, and reflect the pulsed laser to the photodiode.

3.7. Turn on the oscilloscope and then set its trigger threshold to a voltage sufficient to signal from the photodiode.

NOTE: The threshold value may be set smaller than the peak of the trigger signal. When an unintentional reflected light enters the photodiode, the oscilloscope displays a frequency that differs greatly from the pulsed laser frequency. In this case, repeat step 3.6.

3.8. Check the trigger frequency with an oscilloscope and tune the oscillator exactly.

3.9. Put the laser mode on standby.

3.10. Connect a Schottky barrier diode in a microwave waveguide for reflected microwave detection and a signal input channel of the oscilloscope, through a BNC cable.

3.11. Apply a 9.5 V operating voltage to a Gunn diode.

3.12. Place the quartz cell (step 2.2) on the stand in front of the aperture as close as possible. Fix with tape.

4. Measurement and saving data

4.1. Turn on the laser light oscillation and irradiate the light to the sample.

4.2. Place a half-waveplate (λ/2), a polarizer, and a power meter on the optical path (**Figure 1**).

4.3. Irradiate the pulsed laser to the power meter, as shown in **Figure 1**. Check the excitation intensity of the laser.

4.4. Adjust the λ/2 angle for control of the excitation intensity.

NOTE: λ/2 changes the polarization direction of the laser light, while the polarizer transmits only one light polarization direction, through which excitation intensity is controlled. Injected photon densities are set to 8×10^{13} cm^{-2} and for the 266-nm laser, the excitation carrier density in 4H-SiC is 4.5×10^{17} cm^{-3}.

4.5. Remove the power meter from the optical path.

4.6. Adjust the time/div and V/div of the oscilloscope so that the peak signal is displayed on the oscilloscope.

4.7. Adjust the amplitude and phase of the microwave through an E–H tuner. Check the oscilloscope and look for the E–H tuner where the peak signal is at maximum. Failed adjustment of the E-H tuner results in signal loss, as depicted in **Figure 2**.

NOTE: An amplifier is used to strengthen the decay signal in case of insufficiently large signal relative to background noise or when it is not observed even after adjusting the E–H tuner. The amplifier is placed between the Schottky barrier diode and the signal input channel of oscilloscope with a BNC cable, as illustrated in **Figure 1**.

4.8. Repeat steps 4.6 and 4.7 to complete the tuning.

4.9. Adjust the time/div of the oscilloscope and sketch a decay curve in the measurement area on the oscilloscope.

4.10. Average the signal for an arbitrary number of times to improve the signal-to-noise ratio.

4.11. Save the measurement data as an electronic file to a memory and then remove it from the oscilloscope.

5. Data processing

5.1. Import the signal data to a personal computer.

5.2. Plot decay curves obtained from the experiment as a function of time.

5.3. Calculate the average value of background noise level, subtract it from the decay signal, and plot it as a function of time.

5.4. Find the peak value of the decay signal obtained in step 5.3 and then divide the decay signal by the peak value.

REPRESENTATIVE RESULTS:

Figure 1 shows a schematic diagram of the μ-PCD apparatus consisting of a 10 GHz microwave frequency, X waveguide band, and a rectangular waveguide. The microwave was focused by the double ridge waveguide and irradiated on the sample. The Gunn diode output power was 50 mW and the phase noise was nearly -80 dBc/Hz.

Figure 3 shows the μ-PCD decay curve of a 100-μm-thick n-type 4H-SiC sample excited on the Si-face by 266 nm in the air; μ-PCD signal (V) scaled logarithmically was the dependent variable and time (μs) was the independent variable. The signal voltage peak was approximately 0.046 V prior to amplification. Moreover, the observed voltage of the direct current (DC) component of the reflected microwave obtained from the oscilloscope DC mode was of the order of several volts. As recombination of excess carriers progressed with time, the sample's conductivity and microwave reflectance decreased.

Figure 4 shows the normalized μ-PCD decay curve of **Figure 3**. Normalization enables comparison of the time constants with different peak intensities. Typically, carrier lifetime

estimation based on the decay curve is carried out with the 1/e lifetime $\tau_{1/e}$ parameter, indicating that time expended to obtain signal intensity decreases from the peak to 1/e (~0.368). Note that μ-PCD decay was not a single exponential and $\tau_{1/e}$ was influenced by both bulk and surface recombination. However, comparing the time constant of samples having different thickness or surface condition necessitated a reference parameter. Usage of $\tau_{1/e}$ was convenient given the good signal-to-noise ratio at the initial part of the decay curve and the simplicity of the data analysis. To characterize the μ-PCD signal, half-time lives, I_{40}/I_{max}, and k_D constant also adopted such parameters[22–24]. In fact, $\tau_{1/e}$ was adopted in the SEMI standard: SEMI MF 1535[8] as the standard for carrier lifetime measurement of Si. For the decay curve in **Figure 4**, $\tau_{1/e}$ was approximately 0.34 μs.

In **Figure 5,** the quartz cell, containing the aqueous solution and with the sample on its wall, was placed on the stand in front of the aperture[11]. Each intensity of the irradiated microwave and the reflected microwave from the sample, as well as the μ-PCD signal-to-noise ratio, were dependent on the distance between the sample and the aperture, which, ideally, should be as close as possible. In the actual measurement, the distance obtained was as close as possible; measurement using the quartz cell yielded a distance of 0.5 mm, which was of the same as the thickness of the quartz cell glass.

Figure 6 shows μ-PCD decay curves of the n-type 4H-SiC in the air and in aqueous solutions. An excitation light of 266 nm was irradiated to the Si-face of the 4H-SiC. Aqueous solutions used had concentrations, as mentioned previously, as follows: 1 M each of H_2SO_4, HCl, Na_2SO_4, or NaOH, or 1 wt% of HF. The time constant of the decay curves was longer with the sample immersed into the acidic aqueous solutions (i.e., H_2SO_4, HCl, or HF), implying that acidic solutions passivated surface states on the Si-face and reduced surface recombination of the excess carriers.

Figure 7 shows the pH dependence of $\tau_{1/e}$ of the n-type 4H-SiC sample excited on the Si-face at 266 nm of light. The pH was calculated from the molar concentrations of H_2SO_4, HCl, and NaOH. This figure indicated the carrier lifetime dependence on pH aqueous solutions; therefore, lower pH would have more effects on the carrier lifetime.

Surface recombination velocity S was calculated to reproduce the $\tau_{1/e}$ used for the samples. The decay model of excess carriers has been reported in refs. 2 and 3. To obtain the excess carrier concentration $\Delta n(x, t)$, the following continuity equation was solved. Here, $\Delta n(x, t)$ was defined as a function of time t and depth x in a semiconductor layer; thus,

$$\frac{\partial \Delta n(x,t)}{\partial t} = D_a \frac{\partial^2 \Delta n(x,t)}{\partial x^2} + \frac{\Delta n(x,t)}{\tau_B} + B\Delta n^2 + C\Delta n^3, \qquad (6)$$

where τ_B is bulk lifetime due to the Shockley–Read–Hall (SRH) recombination, D_a is the ambipolar diffusion coefficient, B is the radiation recombination coefficient, and C is the Auger recombination coefficient.

At the excited and other surfaces, boundary conditions were given by Equation 7:

$$D_a \frac{\partial \Delta n(0,t)}{\partial x} = S_0 \Delta n(0,t) \text{ and } D_a \frac{\partial \Delta n(W,t)}{\partial x} = S_W \Delta n(W,t) \qquad (7)$$

where S_0 and S_W denote the surface recombination velocity of the excited and other surfaces, respectively, and W is the layer thickness.

Moreover, the initial excess carrier concentration profile using light pulse illumination could be expressed using Equation 8:

$$\Delta n(x, 0) = g_0 \exp(-ax) \quad (8)$$

where g_0 is carrier concentration at x = 0 and α is the absorption coefficient.

Solving Equation 6 by employing the boundary conditions of Equation 7 and the initial condition of Equation 8 provided the excess carrier decay curves. In the process, S was estimated by comparing the $\tau_{1/e}$ obtained from the experiments and from the calculated decay curves. Least squares fitting minimized errors between the experimental $\tau_{1/e}$ in the all conditions and the calculated $\tau_{1/e}$ with parameters S_0, S_w, and τ_B.

As depicted in Equation 6 carrier recombination is the summation of various decay components, namely, surface, SRH, radiative, and Auger recombinations, the last two having remarkable high carrier density. On the other hand, SRH recombination depends on point defects and dislocations in the bulk of the semiconductor material that form energy levels in the semiconductor band gap. The energy levels act as stepping stones for carrier transition between the valence and conduction bands.

μ-PCD also shows nonlinearity at a high injection condition, and overestimates carrier lifetime[13,25,26]**. Figure 8** shows the measured μ-PCD under a high excitation condition. Note that the decay curve for an injected photon density of 10^{15} cm^{-2} became more gradual compared to that for photon density of 10^{14} cm^{-2}, owing to microwave nonlinearity. Furthermore, the measurement examples shown in **Figure 3, Figure 4,** and **Figure 6** were obtained for an injected photon density of 8 x 10^{13} cm^{-2} resulting in negligible microwave nonlinearity, and Auger and radiative recombinations but dominant SRH and surface recombinations.

Figure 6 can be taken to exemplify decay curve calculations for the n-type 4H-SiC Si-face excited by 266 nm light, by referring to the dashed lines, where τ_B = 3 μs and S for the Si-face S_{Si} = 200 cm/s or 700 cm/s. For both S_{Si} settings, the experimental decay curve measured in neutral pH (air, 1 M Na_2SO_4) and in the acidic condition (1 M H_2SO_4), respectively, were well reproduced, which meant that S_{Si} for the n-type 4H-SiC significantly reduced from 700 cm/s to 200 cm/s in acidic aqueous solutions as hydrogen passivated the surface states on the Si-face.

FIGURE LEGENDS:

Figure 1: Schematic diagram of the μ-PCD device. A part of the laser light is reflected by the half-reflection mirror. The reflected laser is detected by the photodiode, and a signal coming from the photodiode is used as a trigger for the oscilloscope. A microwave is generated from the Gunn diode in the direction bent by the circulator; then, a microwave goes through the aperture and irradiates the sample. The reflected microwave from the sample comes back to the aperture and into the circulator, where it is detected by the Schottky barrier diode. Finally,

the signal coming from the Schottky barrier diode is observed by the oscilloscope.

Figure 2: The µ-PCD signal for a failed tuning of E-H tuner. No measurable peak is observed for a failed tuning.

Figure 3: The µ-PCD decay curve for the n-type 4H-SiC sample with excitation on the Si-face by 266 nm in air. The pulsed laser is irradiated at time = 0 s at which the signal intensity is at maximum. This figure has been modified from Ichikawa et al.[11] with permissions.

Figure 4: Normalized µ-PCD decay curve for the n-type 4H-SiC sample with excitation on the Si-face by 266 nm in air. The maximum value of the decay curve in **Figure 2** is normalized to unity. The value of the dashed line is 1/e, and $\tau_{1/e}$ is approximately 0.34 µs as depicted. This figure has been modified from Ichikawa et al. [11] with permissions.

Figure 5: Image of µ-PCD measurement in an aqueous solution in a quartz cell. The quartz cell is placed on the stand in front of the aperture to allow µ-PCD decay curve measurement in an aqueous solution. The cell dimension is 5 mm x 20 mm x 40 mm (length x width x height).

Figure 6: Normalized and calculated µ-PCD decay curves for the n-type 4H-SiC sample with excitation on the Si-face by 266 nm, in air and aqueous solutions. Solid lines represent the µ-PCD experimental result curves for the aqueous solutions of H_2O, H_2SO_4, HCL, Na_2SO_4, NaOH, or HF. The dashed lines are calculated curves with the bulk carrier lifetime in the epilayers, τ_B = 3 µs, and the surface recombination velocity for the Si-face, S_{Si} = 200 cm/s or 700 cm/s. This figure has been modified from Ichikawa et al.[11] with permissions.

Figure 7: The pH dependence of $\tau_{1/e}$ for the n-type 4H-SiC sample with excitation on the Si-face by 266 nm. Carrier lifetime increases as the pH of the aqueous solution decreases. This result indicates that lower pH will have more effects on the carrier lifetime. This figure has been modified from Ichikawa et al.[11] with permissions.

Figure 8: The µ-PCD decay curve of n-type 4H-SiC with excitation of injected photon density of 10^{14} or 10^{15} cm^{-2} on the Si-face by 266 nm. Measurement with high excitation at photon density of 10^{15} cm^{-2} makes a more gradual decay curve than that with lower photon density due to nonlinearity of microwave reflectivity.

DISCUSSION:

In the µ-PCD protocol, step 4.7 is the most important point. The E–H tuner was incorporated with a movable short circuit in the E and H planes, respectively. Thus, moving the short circuit of the E tuner or the H tuner changes the amplitude and phase of the reflected microwave and maximizes the signal amplitude. Tuning has a large influence on the waveform of the decay curve and must be performed strictly. In case of a weak signal strength where tuning might be difficult, a few tens of tuning averages may be used. For failed tuning, the µ-PCD decay curves are not observable; only the noise signal of an oscilloscope is observed. **Figure 2** shows the oscilloscope waveform in such a case.

It is easy to measure highly resistive samples as there is no lower conductivity limit. When the sample resistivity is low or when the sample is thick, the skin effect of the microwave is not negligible. The distance until the electric field intensity of the microwave becomes 1/e times is referred to as skin depth δ, which is expressed by Equation 9:

$$\delta = \left[\frac{\omega\mu}{2}\left(\sqrt{(\omega\varepsilon)^2 + \rho^{-2}}\right) - \omega\varepsilon\right]^{-\frac{1}{2}} \quad (9)$$

where ω is the angular frequency of the microwave, and ε, ρ, and μ represent the sample's dielectric constant, resistivity, and permeability, respectively. In the case of Si and SiC, approximate δ values for the 10 GHz microwave were 9 mm at 50 Ω·cm, 2 mm at 10 Ω·cm, 500 μm at 1 Ω·cm, and 150 μm at 0.1 Ω·cm. Therefore, measurements for samples with typical thicknesses (several hundred micron) at less than 0.1 Ω·cm will lose δ accuracy. On the other hand, the microwave and optical radiation are incident from the opposite of the wafer in this protocol. A negligible skin effect indicates better microwave and optical radiation from the same side.

Lower limits depend on the resistivity and thickness of the sample resulting from its interaction with the microwave. For highly resistive samples, the typical lower limits of the excess carriers are on the order of 10^{12} cm^{-3}. On the other hand, electron–hole scattering must be considered at excess carriers greater than 10^{16} cm^{-3}, as discussed in ref. 13.

The μ-PCD decay curves became gentle at high excitation density due to unproportionality of the microwave reflectivity to the excess carrier concentration such that Equation (3) would lose its validity[13,25,26] and $\tau_{1/e}$ would be overestimated. **Figure 8** shows the μ-PCD decay curve of a chemical mechanical polishing surface treatment n-type 4H-SiC with excitation on the Si-face by 266 nm under high excitation intensity.

Moreover, time resolution depends on the performance of the measurement apparatus such as an excitation source, an oscilloscope, and an amplifier. For example, in this study, the apparatus consisted of a pulsed laser with pulse width of 1 ns as the excitation source and an oscilloscope having a frequency band of 500 MHz. Consequently, the minimum measurable lifetime was estimated at 2 ns.

As mentioned earlier, μ-PCD is very useful for characterization of semiconductors such as Si. Nevertheless, its application can be extended to other materials, for instance, in photoactive materials including TiO_2[27–30].

Furthermore, aside from the μ-PCD, TR-PL[2] and TR-FCA introduced at the earlier sections are the other two carrier lifetime measurement techniques. TR-PL observes the time change of photoluminescence caused by carrier recombination while TR-FCA observes the time change of probe light absorption[4]. Specifically, free carrier absorption occurs when light with energy smaller than the band gap is irradiated during carrier excitation[3]. Nevertheless, compared to these two, μ-PCD directly observes electrical conductivity by microwave and has a high surface roughness and signal sensitivity, making it the more ideal method for carrier lifetime

measurement for semiconductor device applications.

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DISCLOSURES:

The authors have nothing to disclose.

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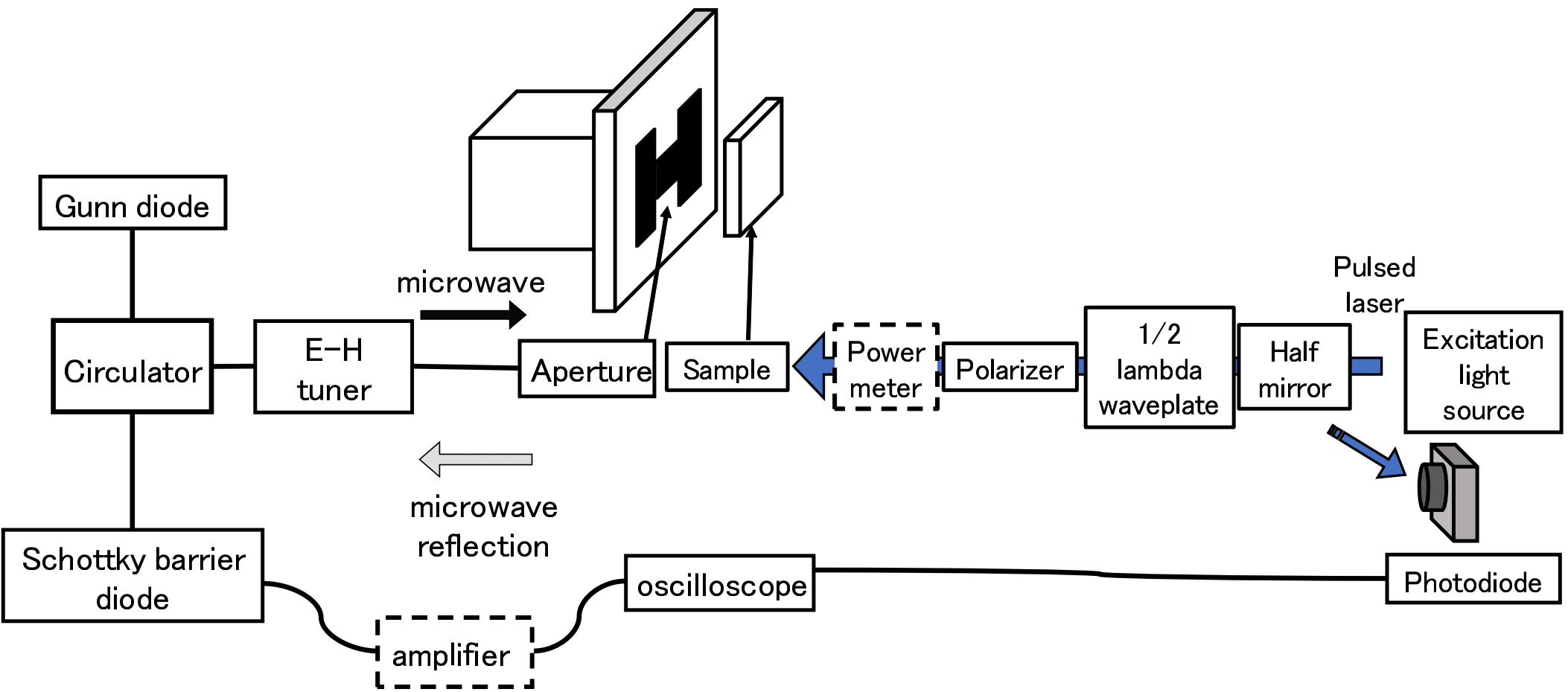

Figure2revised

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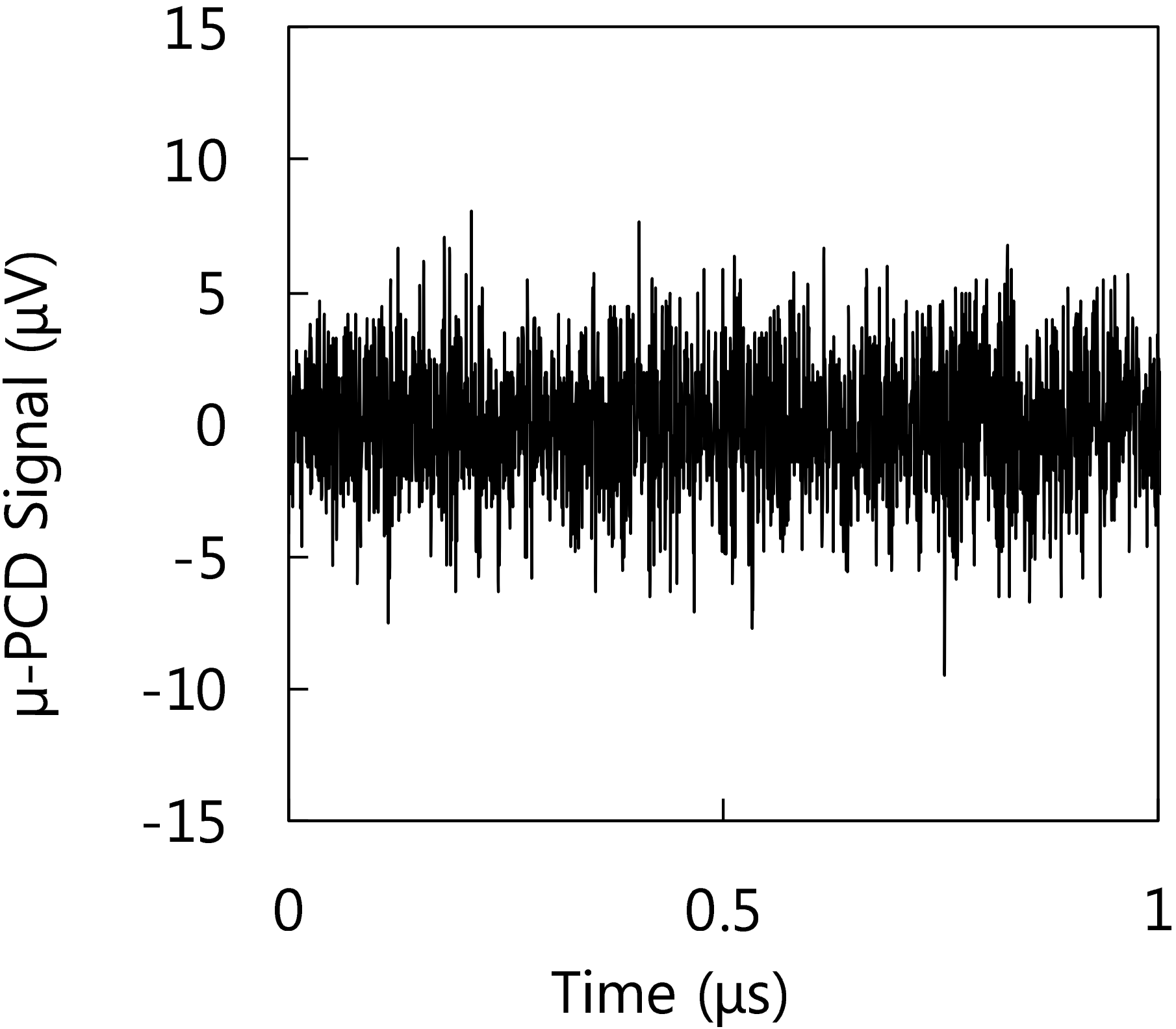
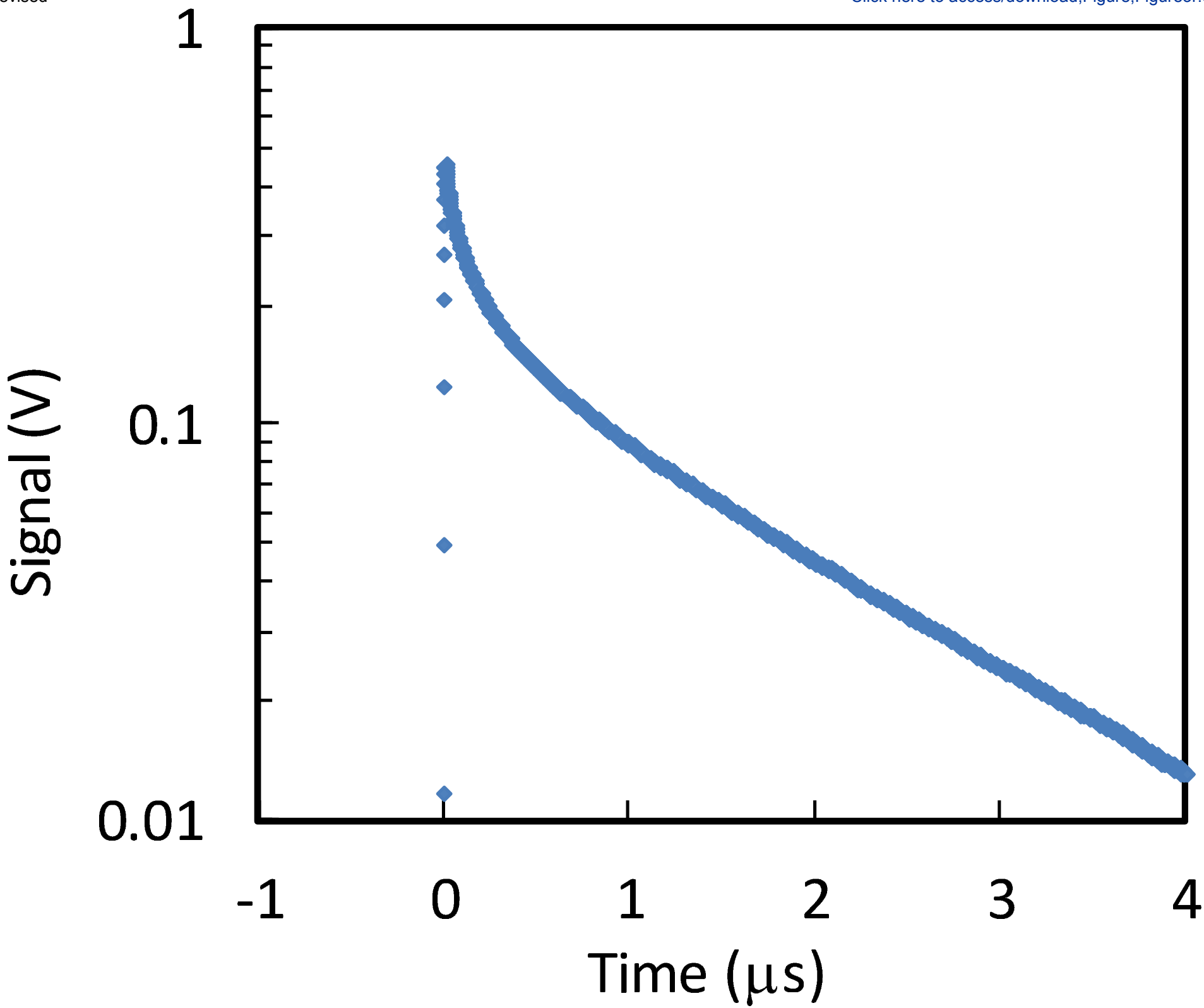
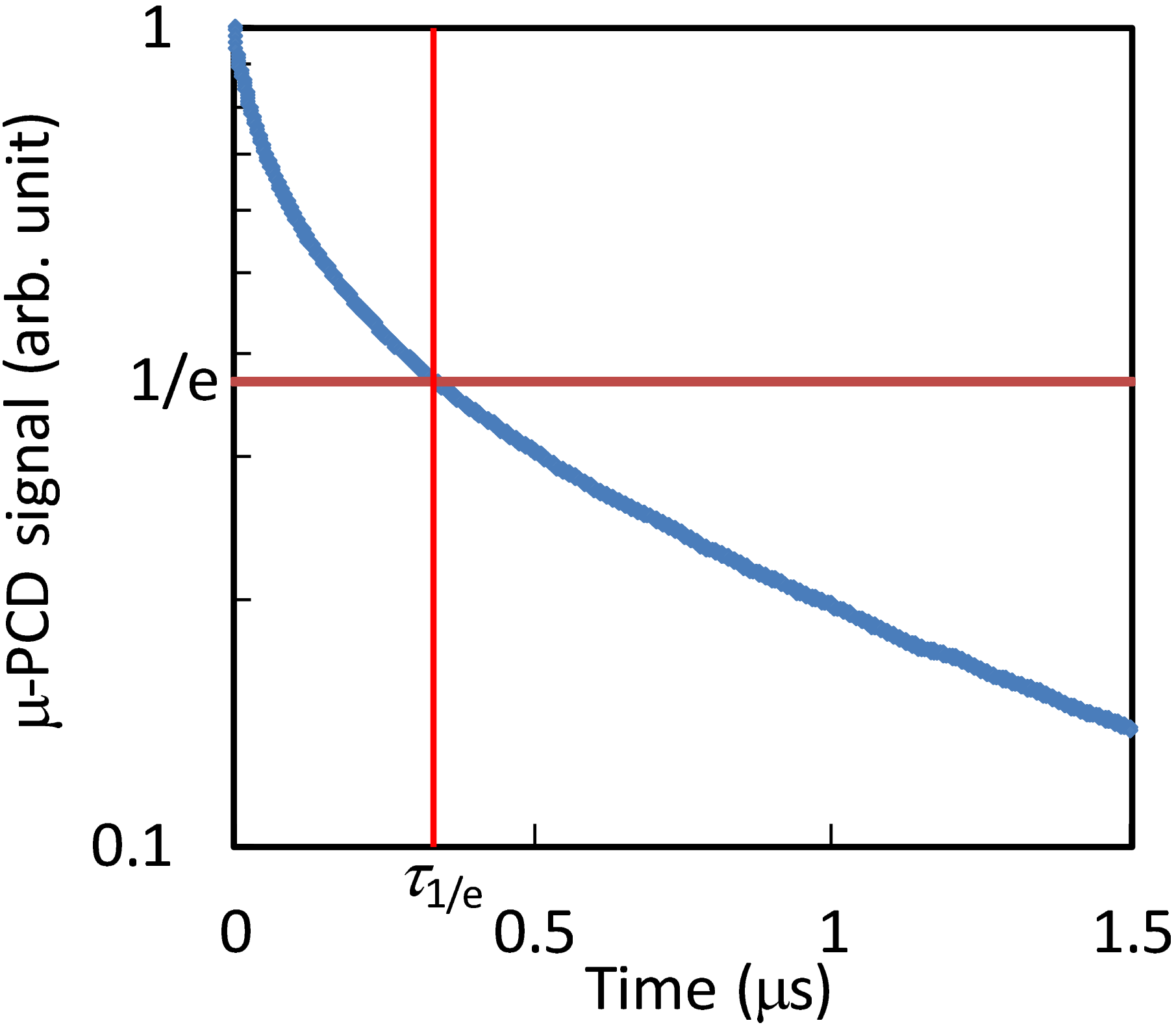
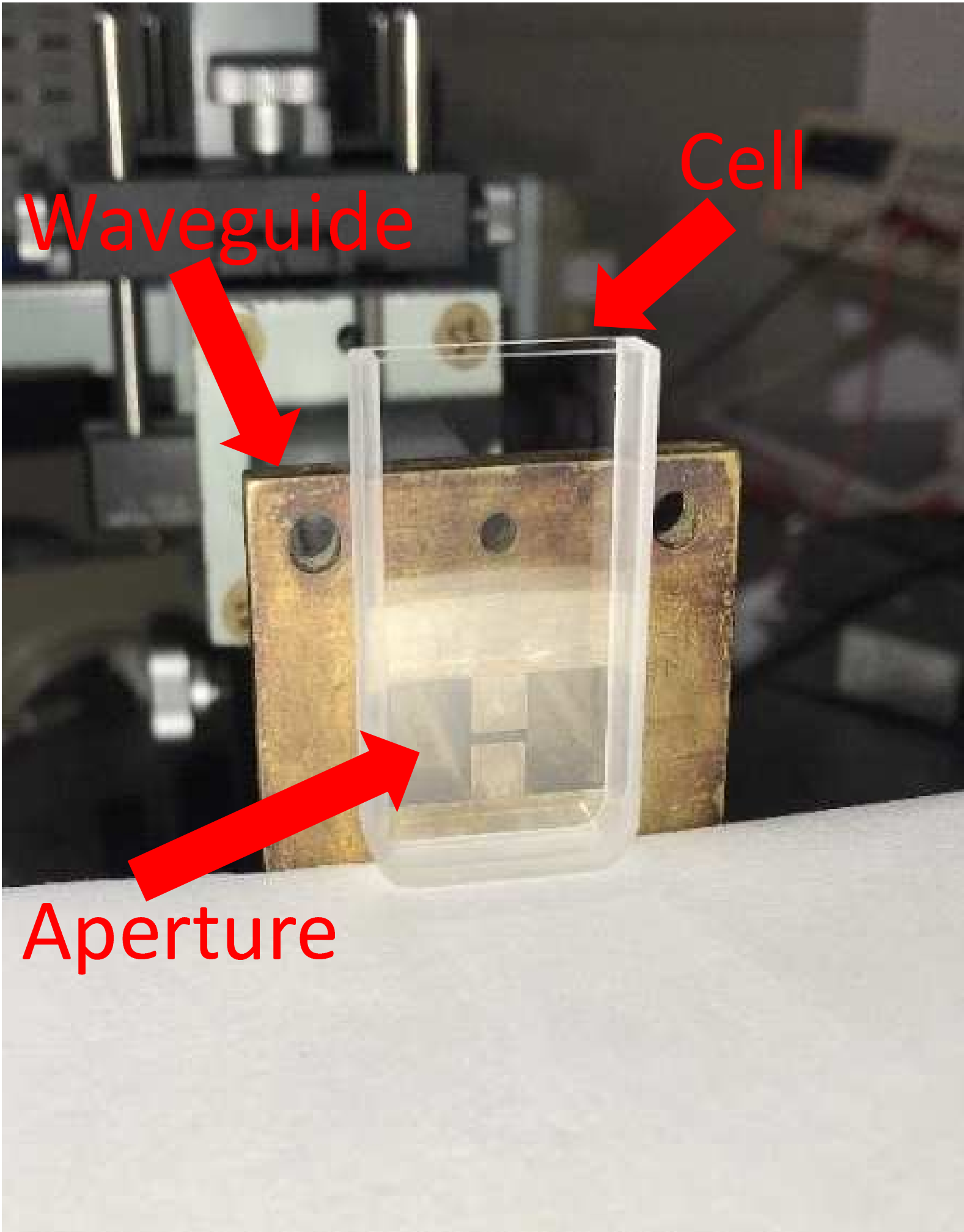


Figure3revised

Click here to access/download;Figure;Figure3r.eps







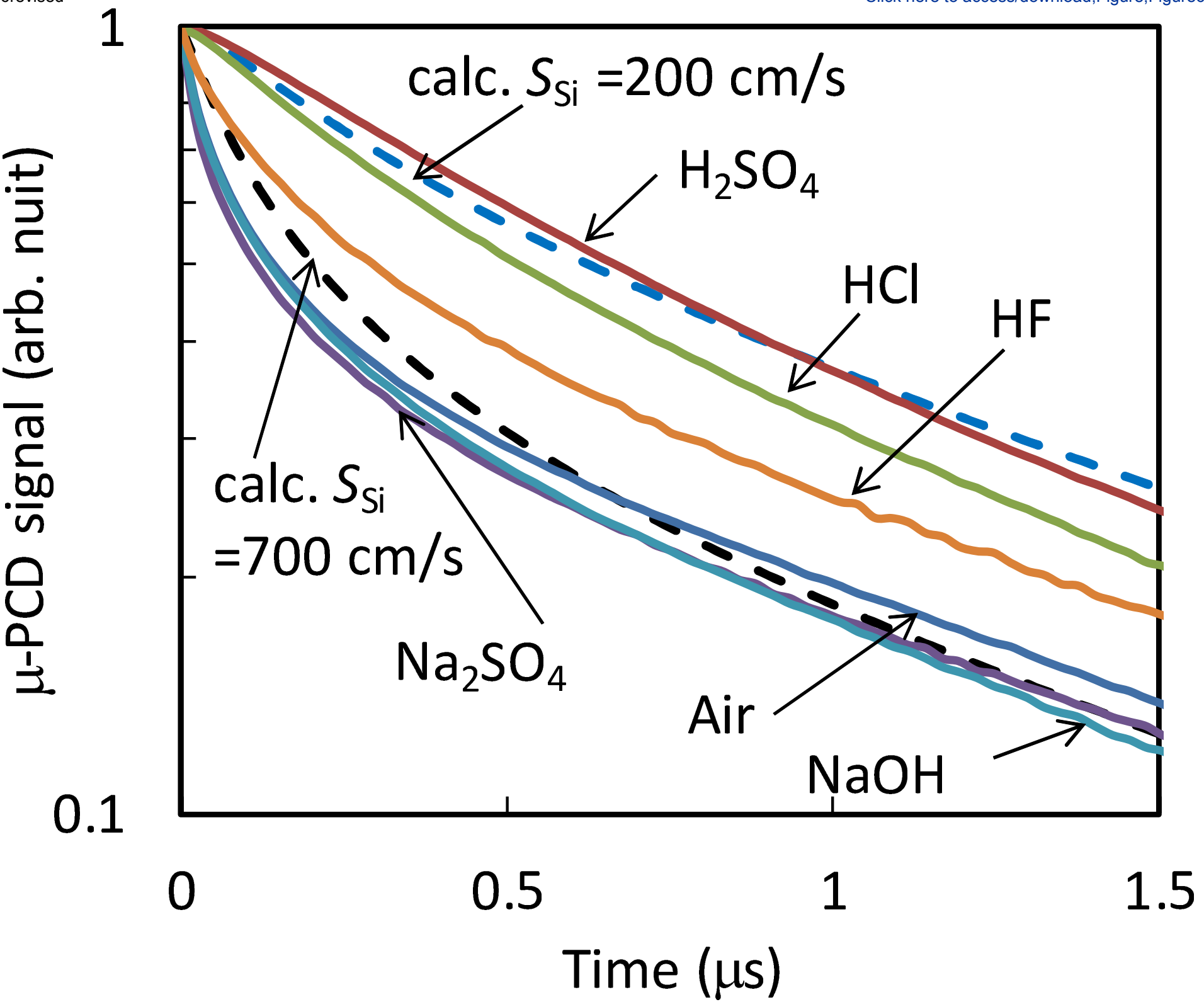


Figure7

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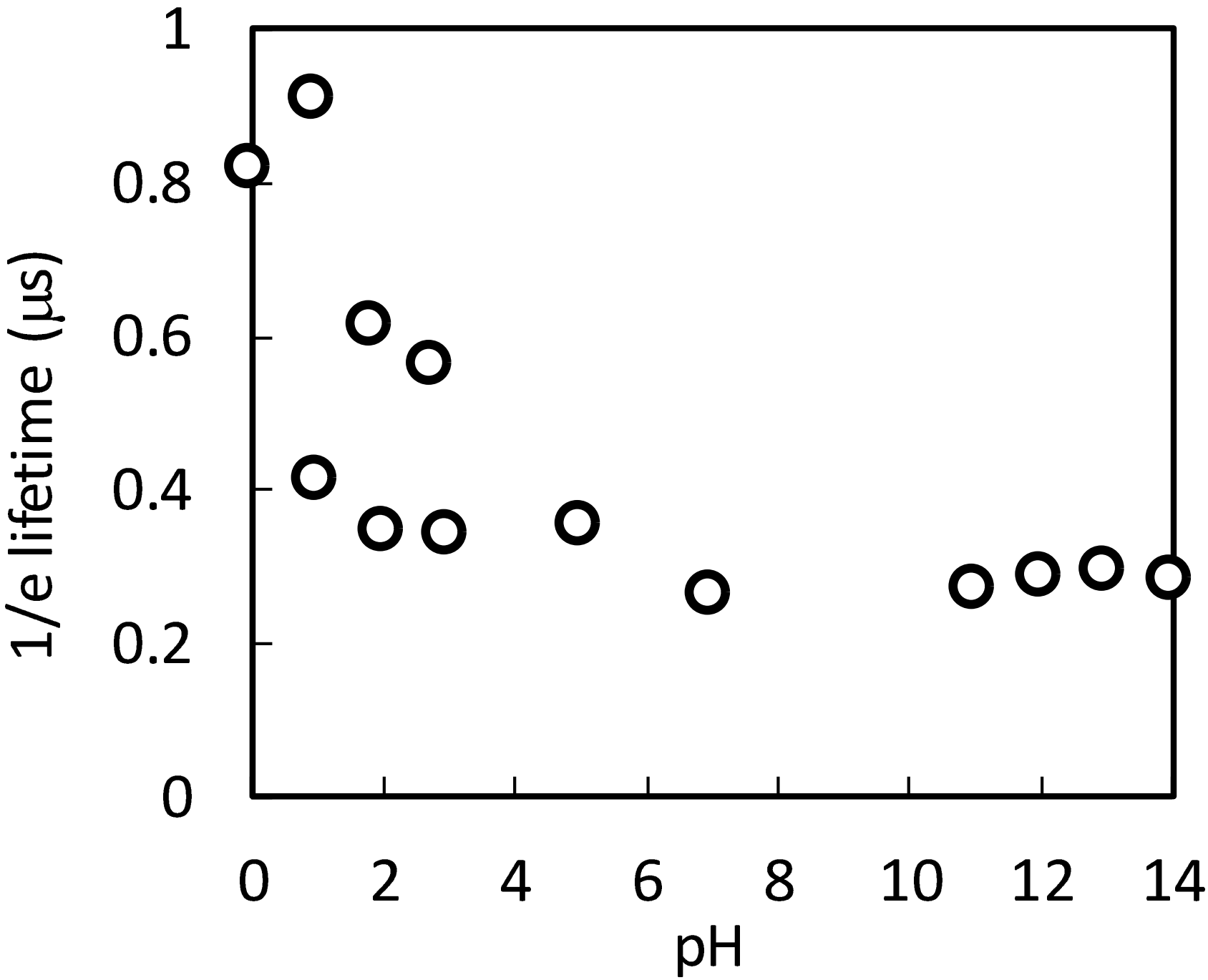
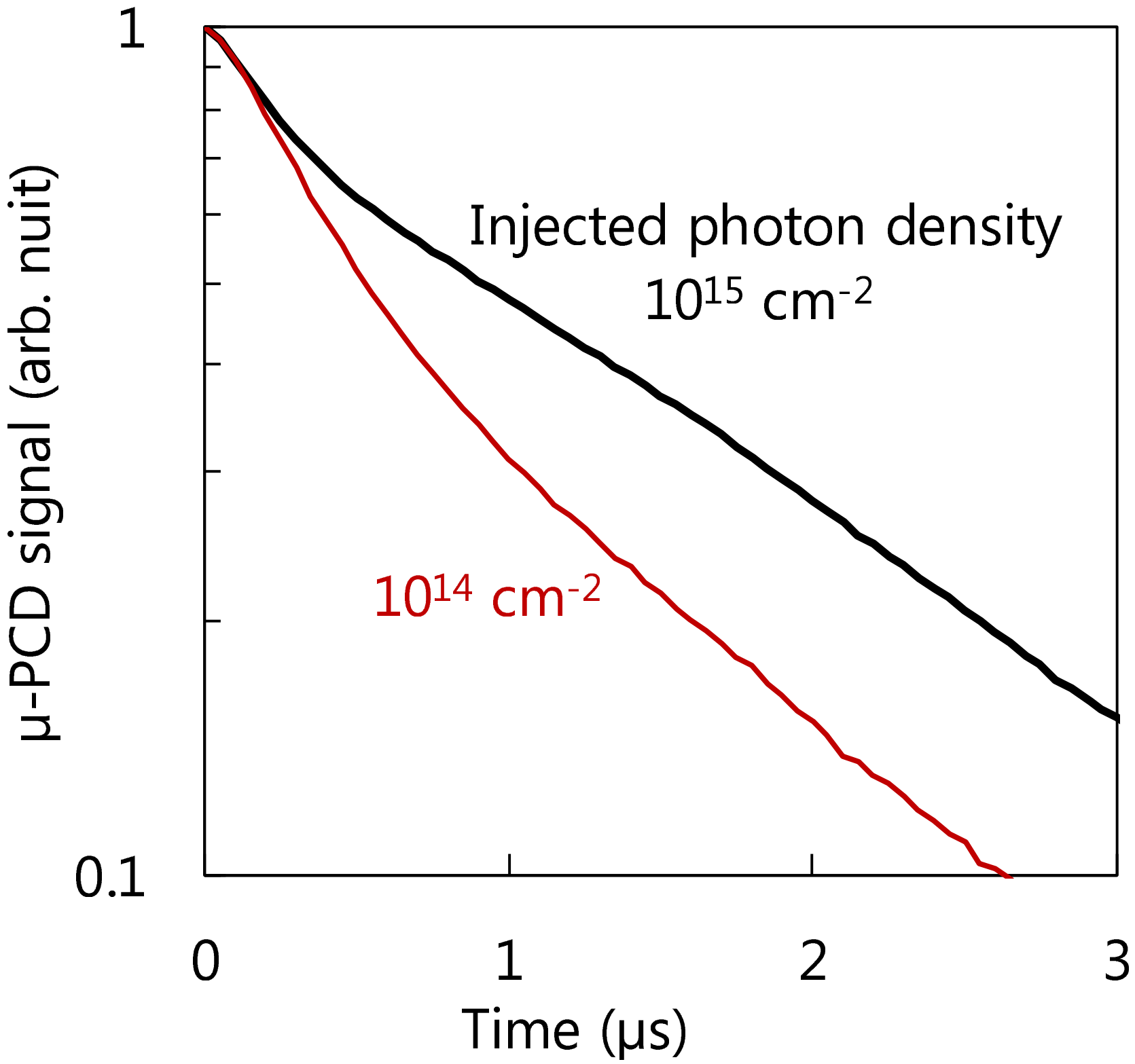


Figure8

Click here to access/download;Figure;Figure8.eps



Name of Material/ Equipment	Company	Catalog Number
n-type 4H-SiC epilayer	Ascatron AB http://ascatron.com/	
266 nm pulsed laser	CryLaS GmbH http://www.crylas.de/	FQSS 266-50
Photodiode	THORLABS https://www.thorlabs.com/index.cfm	DET10A/M
Schottky barrier diode	ASI http://www.advancedsemiconductor.com/	1N23WE
Gun diode	Microsemi https://www.microsemi.com/	MO86751C
E-H tuner	SPC ELECTRONICS CORPORATION http://www.spc.co.jp/index.html	
Circulator	SPC ELECTRONICS CORPORATION http://www.spc.co.jp/index.html	
Rectangular waveguide	SPC ELECTRONICS CORPORATION http://www.spc.co.jp/index.html	
Double ridge waveguide	SPC ELECTRONICS CORPORATION http://www.spc.co.jp/index.html	
Crystal mount	SPC ELECTRONICS CORPORATION http://www.spc.co.jp/index.html	
Acetone	KANTO CHEMICAL CO.,INC. https://www.kanto.co.jp/	GE00001
Sulfuric acid	KANTO CHEMICAL CO.,INC. https://www.kanto.co.jp/	GE00257
Hydrochloric acid	KANTO CHEMICAL CO.,INC. https://www.kanto.co.jp/	GE00238
Hydrogen fluoride	KANTO CHEMICAL CO.,INC. https://www.kanto.co.jp/	18083-1B
Sodium hydroxide	KANTO CHEMICAL CO.,INC. https://www.kanto.co.jp/	37184-00
Sodium sulfate	KANTO CHEMICAL CO.,INC. https://www.kanto.co.jp/	37280-00

Comments/Description
Sample
Excitation light source
Trigger signal detection
Reflected microwave detection
Microwave generation source
microwave component
microwave component
microwave component
microwave component
microwave component
Sample cleaning
Acidic aqueous solution
Acidic aqueous solution
Acidic aqueous solution
Alkaline aqueous solution
Neutral aqueous solution

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Author(s): Takato Asada, Toshihito Ichikawa, Masashi Kato

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Title:	Associate Professor
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Thank you for inviting us to submit a revised draft of our manuscript entitled, JoVE59007 to Journal of Visualized Experiments. We also appreciate the time and effort you and each of the reviewer have dedicated to providing insightful feedback on ways to strengthen our paper. Thus, it is with great pleasure that we resubmit our article for further consideration. Please find the revised manuscript and the revised or added texts were green colored. We have incorporated changes that reflect the detailed suggestions you have graciously provided. We also hope that our edits and the responses we provide below satisfactorily address all the issues and concerns you and the reviewers have noted.

Editorial comments:

1. Please employ professional copy-editing services as the language in the manuscript is not publication grade. There are many awkward phrases and missing articles throughout.

Thank you for pointing out.
We employed professional copy-editing services, and we proofread the sentences.

2. Please revise lines 211-212, 220-221, 229-232, 234-235, 241-248 to avoid previously published text.

The line was changed as the sentence was revised:(line 211-212 to 219-221, 220-221 to 237-238, 229-232 to 250-253, 234-235 to 255-256, 241-248 to 259-267).
We revised these texts to avoid previously published text.

3. Please revise the title to be more concise.

We changed the title of this paper to Protocol of Carrier Lifetime Measurements in Semiconductors through the Microwave Photoconductivity Decay Method

4. Please expand your Introduction to include the following: The advantages over alternative techniques with applicable references to previous studies; Description of the context of the technique in the wider body of literature; Information that can help readers to determine if the method is appropriate for their application.

Thank you for advises. We expanded Introduction to include the advantages over alternative techniques with applicable references to previous studies with reference.
We added line 51-63 and references.

5. 1.1: Please describe how to prepare the n-type 4H-SiC epilayer. If it is purchased, please provide product information in the Table of Materials.

The n-type 4H-SiC epilayer is product of Ascatron AB. It is a custom made product. We added it to the material list.

6. 2.1: What volumes of these solutions are needed?

The sample was immersed solution in the quartz cell. Therefore, we need an aqueous solution in which the entire sample can be immersed. When using this quartz cell, at most 4 ml of solution is required.
We corrected line 102-104.

7. 2.2: This step is unclear. Is the sample or the quartz cell containing the sample dipped in the aqueous solution? What does the aqueous solution stand for, a mixture of 1 M H_2SO_4, 1 M HCl, 1 M Na_2SO_4 and 1 M NaOH, and HF? Please clarify.

I apologize for the mistake of the paper I submitted last time. I incorrectly entered the size of the quartz cell. In fact, the size of quartz cell is Length 5 mm x Width 20 mm x Height 40 mm. These aqueous solutions (1 M H_2SO_4, 1 M HCl, 1 M Na_2SO_4,1 M NaOH, or HF at concentration of 1 wt %) are not mixed. Choose one aqueous solution for measurement. Pour the solution into the quartz cell and dip the sample in the aqueous solution.We corrected line 95-96, 198-100 and 323.

8. 3.2: What does BNC stand for? Please spell it out.

BNC is an abbreviation for Bayonet Neill Concelman.
We corrected line 111.

Reviewer #4:

(1) Some results for de-turning of E-H tuner are helpful.

If tuning of E-H tuner failed, the μ-PCD decay curves is not observed but only noise signal is observed with an oscilloscope. We added Figure 2 (newly numbered) which shows the waveform of oscilloscope when tuning of E-H tuner is failed.
We added line 170-171 and 307-308.

(2) Some results under higher excitation density are helpful.

Under higher excitation density, Auger and radiative recombination will not be negligible. In addition, μ-PCD decay curves becomes gradual because microwave reflectivity is not proportional to the excess carrier concentration and Eq. (3) lost validity[13,25,26]. As a result, $\tau_{1/e}$ is overestimated under higher excitation density condition. **Figure 8** shows μ-PCD decay curve of the chemical mechanical polishing (CMP) surface treatment n-type 4H-SiC with excitation on the Si-face by 266 nm under high excitation intensity.

(3) As the authors mentioned at L217, the distance between the sample and the aperture is important for reliable evaluation. The authors should explain how to adjust the distance for actual measurements.

The distance between the sample and the aperture should be as close as possible, because it has a great effect on the signal-to-noise ratio of the signal. In actual measurement, the distances of them is as close as possible. In the measurement using a quartz cell, the distance of them is 0.5 mm the thickness of the glass of the quartz cell.
We added line231-235.

(4) Physical meaning of $\tau_{1/e}$ is unclear because decay profiles are not single exponential.

1/e lifetime $\tau_{1/e}$ is the time excess carrier decay from a peak to 1/e. As you said, μ-PCD decay is not a single exponential and $\tau_{1/e}$ is influenced by both bulk and surface recombination. However, in order to compare time constant with samples with different thickness or surface condition, an unified criteria is required.

In addition, because the initial part of the decay curve has good signal-to-noise ratio and simplicity of the data analysis, usage of $\tau_{1/e}$ is convenient.
In fact, $\tau_{1/e}$ is adopted in the SEMI standard: SEMI MF 1535[8] as a standard for carrier lifetime measurement of Si. We revised and added line 218-226.

(5) Physical meaning of the bulk lifetime (τ_B) is unclear. The authors should clarify actual reaction processes in semiconductor to determine τ_B.

We defined bulk lifetime as carrier recombination lifetime due to Shockley-Read-Hall (SRH) recombination. We are sorry ambiguous definition in the previous manuscript. SRH recombination depends on point defects and dislocations in bulk of a semiconductor material. This is because that the point defects and dislocations form energy levels in the band gap of the semiconductor. The energy levels act as stepping stones for carrier transition between the valence and conduction bands.
We revised and added line 255 275-280.

Reviewer #5:
-The transient technique using microwave absorption to follow charge carrier dynamics in semi-conductor, mainly developed by J. Warman and M. Kunst, is usually named TRMC (Time Resolved Microwave Conductivity), see Ref. 5 in the text. Some authors are using also TPC (Transient Photo Conductivity). In this work, this technique is called μ-PCD. It is not a problem, but a reference to TRMC should be made, and the authors should clarify their choice.

Thank you for the suggestion. We added sentences at line 51-55.

-TRMC (μ-PCD) is very useful for semi-conductors like Si, but it has been employed a lot to characterize photoactive materials like TiO2. A reference to this application of the method should also been made.
Schindler, K.-M., and Kunst, M. (1990) "Charge-Carrier Dynamics in TiO2 Powders," J. Phys. Chem., 94, pp. 8222-8226.
Savenije, T. J., de Haas, M. P., and Warman, J. M. (1999) "The Yield and Mobility of Charge Carriers in Smooth and Nanoporous TiO2 Films," Zeitschrift für Phys. Chemie, 212, pp. 201-206.

Colbeau-Justin, C., Kunst, M., and Huguenin, D. (2003) "Structural influence on charge-carrier lifetimes in TiO2 powders studied by microwave absorption," J. Mater. Sci., 38, pp. 2429-2437.

Thank you for pointing out. I will expand a description about TiO_2. We added the sentences at line 384-386 and references.

-The TRMC decay is not easily simulated by a simple physical model. Mainly because two or more distinct phenomena occur during the decay. The characterization of the decay with one parameter is not trivial. The choice and the use of $\tau_{1/e}$ parameter should be clarified and simplified. A reference to other used parameters may be useful (half-time lives, I40/Imax, kD constant…).
Kolen'ko, Y. V, Churagulov, B. R., Kunst, M., Mazerolles, L., and Colbeau-Justin, C. (2004) "Photocatalytic properties of titania powders prepared by hydrothermal method," Appl. Catal. B Environ., 54, pp. 51-58.
Carneiro, J. T., Savenije, T. J., Moulijn, J. A., and Mul, G. (2011) "The effect of Au on TiO2 catalyzed selective photocatalytic oxidation of cyclohexane," J. Photochem. Photobiol. A Chem., 217, pp. 326-332.
Luna, A. L., Novoseltceva, E., Louarn, E., Beaunier, P., Kowalska, E., Ohtani, B., Valenzuela, M. A., Remita, H., and Colbeau-Justin, C. (2016) "Synergetic effect of Ni and Au nanoparticles synthesized on titania particles for efficient photocatalytic hydrogen production," Appl. Catal. B Environ., 191, pp. 18-28.

I appreciate your suggestion. As you said, other parameters can be employed and recombination phenomena are not simple to be described by only $\tau_{1/e}$. But in the Si industry, this parameter is commonly used. We described reason for adoption of $\tau_{1/e}$ at line 218 to 226.