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Gridded Ionization Chambers for Time Resolved X-Ray Absorption Spectroscopy

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Abstract. Common parallel plate ionization chambers are bandwidth limited by the ion drift velocity. Therefore they can severely decrease the effective energy resolution of time resolved EXAFS or XANES spectra. We therefore developed gridded ionization chambers which suppress the ionic component of the ionization current, which results in a substantial improvement of its bandwidth of typically two orders of magnitude. The rise time of these chambers is measured to be less than 5.5 µs. We have investigated the step response of parallel plate and gridded ionization chambers and discuss their influence on QEXAFS spectra.

1. Introduction
In recent years, time resolved X-ray absorption spectroscopy, especially QEXAFS (Quick scanning EXAFS [1, 2]), has been further developed [3-6] and has become an indispensable tool for in-situ measurements of solid state and chemical reactions, phase transitions, layer growth and more [7-10].

The achievable time resolution of QEXAFS measurements is mainly limited by the available photon flux, the capabilities of the monochromator, i.e. its maximum scanning speed, and in particular by the response time of the ionization chambers and current amplifiers. The increase in intensity at modern 3rd generation synchrotron radiation sources and the development of specialized monochromators allow acquiring complete EXAFS spectra covering several hundreds of eV within less than one hundred milliseconds, amounting to scan rates of up to 0.1 eVµs⁻¹ [11, 12]. Since the typical rise time of ionization chambers amounts to some hundred microseconds, this will clearly affect the measurement and decrease the effective energy resolution of the spectra [13-15].

2. Principles of parallel plate and gridded ionization chambers
Common ionization chambers usually consist of two separated plane parallel electrodes in a gaseous environment as schematically depicted in the inset of figure 1. Incident X-rays pass between the electrodes and ionize the gas, creating ion pairs [16]. Immediately after their creation, these ion pairs are drawn apart under the action of an electric field which is caused by an applied high voltage across both electrodes, yielding an electrical current.

2.1 Parallel plate ionization chambers
There are two common approaches for obtaining the pulse shape of a single ionization event [17, 18]. One of these considers the deposited energy in the gas due to collisions of the drifting ion pairs. Since the drift motion is sustained by the electric field, this energy is taken from the electric field. As a consequence, to keep the electric potentials of the electrodes constant, the external high voltage power supply has to drive a current which is proportional to the ionization rate.
The other approach is based on induced surface charges on the electrodes, which are caused by a single point charge located in the ionization region. Since this surface charge is a function of the distance between the point charge and the electrodes, an electrical current is induced as the point charge drifts towards the electrodes. For an ion pair consisting of a positively charged ion and an electron with the drift velocities \( \mu^+ \) and \( \mu^- \), respectively, the current pulse develops as given by equation 1, where \( d \) denotes the distance between both electrodes. The current pulse is defined piecewise, since for \( t = t_{e^-} \) the electron has reached its electrode and does not contribute to current pulse any longer. When \( t = t_{i+} \) the ion has finally arrived at the opposite electrode, and the pulse has finished.

\[
I(t) = \begin{cases} 
\frac{e}{d} (\mu^+ + \mu^-) & : 0 < t < t_{e^-} \\
\frac{e}{d} \mu^+ & : t_{e^-} < t < t_{i+} \\
0 & : otherwise 
\end{cases} 
\]  

(1)

Both approaches show that the output pulse starts to develop as soon as the ion pair is created and begins drifting apart. The development of the pulse ends when the last charge carrier has reached one of the electrodes. This obviously infers that the pulse length and as such the ionization chamber bandwidth are dominated by the slowest charge carrier which contributes to the output current. With typical field strength of some kVcm\(^{-1}\), the positively charged ions only gain drift velocities on the order of a few centimeters per millisecond, while the much lighter electrons are roughly 1000 times faster.

2.2 Gridded ionization chambers
In order to increase the bandwidth of ionization chambers it is necessary to prevent the ions from inducing charges onto the collecting electrode. This can be achieved by a metal grid which is placed between the electrodes and is kept at an appropriate intermediate electric potential, as shown in the inset of figure 2 [18, 19, 20]. The grid screens the electrode to which the output is connected from charge carriers in the ionization region and thusly prevents them from inducing charges. Only, the electrons which are able to pass the grid will contribute to the output pulse.

In the case of a gridded ionization chamber where only electrons are collected equation 1 has to be replaced by equation 2. The distance between the grid and the collecting electrode is denoted by \( d_{\text{Grid}} \).

As soon as the electron reaches the grid at \( t = t_{\text{Grid}} \) the current pulse starts to develop.

\[
I(t) = \begin{cases} 
\frac{e}{d_{\text{Grid}}} \mu^- & : t_{\text{Grid}} < t < t_{e^-} \\
0 & : otherwise 
\end{cases} 
\]  

(2)

3. Results
The convolution of a step function with equation 1 or equation 2 yields the theoretical expectation of the step response of a parallel plate or gridded ionization chamber, respectively. In the case of a parallel plate ionization chamber this convolution yields a linear increase of the current with time which can be segmented into two domains. One where both species of charge carriers contribute to the current and the adjacent where only the ions remain to cause a current. The slope of the linear rise is correlated to the drift velocities of the electrons and ions.

The step response was measured experimentally at the bending-magnet beamline X1 at HASYLAB (DESY, Germany) by means of a fast X-ray chopper which is based on a rotating disc (120 Hz). The monochromatic X-ray beam (9000 eV) was shaped using a slit system (200 µm × 2 mm) between the ionization chamber and the chopper, which in this setup exhibited a rise time of 3.8 µs. The output ionization current is amplified using a newly developed self-made current amplifier and is sampled at 500 kHz with 16 bit resolution. This current amplifier was particularly chosen due to its exceptionally
3.1 Step response of a parallel plate ionization chamber

The parallel plate ionization chamber was equipped with two rectangular shaped copper electrodes (30 mm × 130 mm) separated by 12.5 mm. The chamber was filled with Argon at 1000 mbar resulting in an X-ray absorption of 90%. The ionization chamber was aligned so that the beam entered midway between the electrodes. The step response was measured for different applied voltages and is shown in figure 1. By changing the voltage, the ion drift velocity is altered. This is observable in the change of the slope adjacent to the short instant rise which originates from the fast electrons. The total rise time is on the order of 700 to 1400 microseconds and is clearly dominated by the ion drift motion.

3.2 Step response of a gridded ionization chamber

The same setup was used to measure the step response of a gridded ionization chamber which features an additional grid electrode made of parallel, equally spaced (2 mm) copper wires with a diameter of 0.28 mm. The grid was mounted 5.5 mm above the collecting electrode and 14 mm below the high-voltage-electrode, thus giving roughly the same ionization region as used in the configuration of the parallel plate ionization chamber. A negative potential of 300 V was applied to the grid, and the upper electrode was set to -700 V. The step response, shown in figure 2, does not exhibit the distinct kink which is observable in the plane parallel electrode configuration (c.f. figure 1). This is a clear indication that no ions actually contribute to the ionization current. As a consequence, the rise time is significantly shorter and is determined to be 5.5 µs ± 0.1 µs. Since the observable step response is in fact a convolution of the response behaviors of the chopper and current amplifier, the actual rise time of the ionization chamber is even smaller. Considering the transient time of only the electrons, the rise time is expected to be less than 1 µs.

Figure 1. Step response of the parallel plate ionization chamber measured with different applied voltages. The curves are shifted vertically for better visualization.

Figure 2. Step response of the gridded ionization chamber. The upper electrode is connected to -700 V and the grid to -300 V. The bottom electrode is grounded through the amplifier.
4. Discussion
To meet the increasing demand in time resolution and data quality regarding QEXAFS measurements, specialized high speed ionization chambers are of utmost importance. According to equation 1 and the measurements of the parallel plate chamber the response behavior can be improved by increasing the applied voltage or minimizing the gap of the electrodes. However, to attain rise times on the order of a few microseconds several thousand volts and electrode distances of few millimeters only would be necessary. This, however, is not feasible due to the limited dielectric strength of most filling gases like argon. By inserting a grid electrode to the ionization chamber, the rise time can significantly be improved, without increasing the field strength. The rise time of the gridded ionization chamber was determined to be 5.5 µs ± 0.1 µs, which is still limited by the response time of the current amplifier. This is roughly 2 orders of magnitude faster than comparable parallel plate ionization chambers.

Using this combination of amplifier and ionization chamber, QEXAFS measurements with scan rates of up to 0.5 eVµs⁻¹ should be possible without risking a loss of information due to a reduced energy resolution of the EXFAS or XANES spectra.

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References
[1] Frahm R 1988 *Nucl. Instrum. Methods Phys. Res.* 270 578
[2] Frahm R 1989 *Rev. Sci. Instr.* 60 2515
[3] Richwin M, Zaeppe R, Lützenkirchen-Hecht D and Frahm R 2002 *Rev. Sci. Instr.* 73, 1668
[4] Stötzel J, Lützenkirchen-Hecht D and Frahm R 2010 *Rev. Sci. Instrum.* 81 073109
[5] Stötzel J, Lützenkirchen-Hecht D and Frahm R 2011 *J. Synchrotron. Rad.* 18 165
[6] Fonda E, Rochet A, Ribbens M, Barthe L, et al. 2012 *J. Synchrotron. Rad.* 19 147
[7] Grunwaldt J-D, Beier M, Kimmerle B, Baiker A, et al. 2009 *Phys. Chem. Chem. Phys.* 11 8779
[8] Singh J,Nachtegaal M, Alayon E M C, Stötzel J et al. 2010 *Phys. Chem. Chem. Phys.* 2 653
[9] Reißmann S, Stötzel J, Frahm R, Kleist W, et al. 2011 *J. Am. Chem. Soc.* 133 3921
[10] Stötzel J, Frahm R, Kimmerele B, Nachtegaal M et al. 2012 *J. Phys. Chem. C* 116 599
[11] Frahm R, Grisebock B, Richwin M and Lützenkirchen-Hecht 2003, *AIP Conf. Proc.* 705 1411
[12] Frahm R, Nachtegaal M, Stötzel J, Harfouche M, et al. 2010 *AIP Conf. Proc.* 1234 251
[13] Stötzel J 2008 Diploma thesis, Bergische Universität Wuppertal, Germany
[14] Müller O 2010 Ionization Chambers for Time Resolved X-ray Absorption Spectroscopy
  Diploma thesis, Bergische Universität Wuppertal, Germany
[15] Stötzel J 2012 New methods and applications in time-resolved X-ray absorption spectroscopy,
  ISBN: 978-3-8440-0908-8 Shaker
[16] Pettifer R F, Borowski M and Loeffen P W 1999 *J. Synchrotron. Rad.* 6 217
[17] Knoll G F 2000 Radiation Detection and Measurement 3rd Edition *John Wiley & Sons Inc.*
[18] Wilkinson D H 1950 Ionization Chambers and Counters *Cambridge Monographs on Physics*
[19] Frisch O 1944 *British Atomic Energy Report* BR-49
[20] Bunemann O, Cranshaw T E and Harvey J A 1949 *Canad. J. Res. Sec. A* 27 191