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Enhanced time resolution with a room-temperature energy dispersive X-ray PIN photodiode detector for improved EDX-EELS coincidence measurements in an electron microscope

Although energy dispersive X-ray spectroscopy (EDX) and energy electron loss spectroscopy (EELS) can be acquired simultaneously with a transmission electron microscope (TEM), it is not a common practise. Most users do one at the time despite that the two data streams complement each other very well allowing to have both the high selectivity of EDX with the high energy resolution of EELS. By time correlating simultaneously acquired X-ray events to electron events one can distinguish actual scattering-process correlated electron-X-ray events from unrelated background events [1]. This is extremely useful to improve sensitivity of EELS to trace elements in a matrix (e.g. nitrogen vacancies in diamond) as the subtle edges of the trace elements are otherwise buried in the background noise.

Currently coincidence measurements in a TEM are however limited to low signal to noise ratios by the limited time resolution of the commercial silicon drift detectors (SDDs) used to detect the X-rays [2]. Those are column-mounted and evolved to large sizes to maximise collection efficiencies at the cost of time resolution due to long unsystematic drift times of the signal charge carriers created upon X-ray detection [3]. In order to improve this we are building a proof-of-concept detector for this application. Our approach consists of having a small fully-depleted Si-PIN photodiode detector mounted on the sample holder itself. Its small size ensures neglectable drift times, low capacitance and therefore high acquisition rates and high temporal resolution. Lastly having the sample close to the photodiode preserves collection efficiency.

So far several prototypes have been made and tested in a scanning electron microscope (SEM). Over the different iterations the signal amplifying electronics, noise levels and form factor have been improved. The latest version (see picture) has a low-noise charge-sensitive amplifier circuit with a reverse biased, low capacitance Si-PIN photodiode that is commercially available and inexpensive to replace. The front end has already physical dimensions that would later fit a TEM holder. A theoretical noise analysis has been made and matches nicely to corresponding experimental measurements. Due to relatively high thermal energy of the photodiode, a dc leakage current is formed that substantially increases the dead time of the detector due to an increased reset rate of the charge-sensitive preamplifier. In order to solve this while preventing tedious cooling (cryogenic or peltier cooling) a precisely controlled compensating current is used that proved to work well without elevating noise levels. When irradiating with Cu X-rays the measured pulses exhibit sub-50ns rise times suggesting already a tenfold improvement in time resolution. Initial EDX spectra show an energy resolution of around 1,6keV at 8,05keV (Cu K-α) which we are now trying to improve further to be useful for EDX.

In conclusion, we present the development of an in-house build room-temperature PIN photodiode X-ray detector to improve time resolution and allow for advancements in EDX and EELS coincidence experiments in electron microscopes that so far have been hampered by the slow drift mechanism in SDD setups.

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[1] P. Kruit et al. ; Ultramicroscopy, 13 (1984)
[2] G. F. Knoll, Radiation Detection and Measurement, 2010
[3] D. Jannis et al. ; Appl. Sci. 11, 9058 (2021)

Workshop topics

Detector systems

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