

Hunting the Silicon loss function with 13 keV electrons in a TEM

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Nearly 0.5 million publications in physical science focus on Silicon since 1833 [1]. Approximately 70.000 investigate the optical properties theoretically and experimentally. About 400 of them employ electron energy loss spectrometry (EELS). So, what is the reason for a further investigation of the optical properties of Silicon? The answer is twofold: (a) modern experimental and theoretical methods provide a better accuracy and (b) the published results disaccord up to a certain degree until now. One has still the feeling that the true optical behaviour of Silicon is still not described within a high accuracy level.

The advantages over optical methods when using electron beam techniques for the determination of the optical properties are the higher spatial resolution and the much larger energy range being explored. Nevertheless, such experiments suffer from the excitation of Čerenkov losses and light guiding modes [2]. These additional losses, which are only due to the fact that the swift electrons travel faster than the speed of light inside the Silicon specimen alter the EELS spectrum such that the determination of optical properties by means of Kramers Kronig Analysis (KKA) becomes difficult and questionable. Therefore the energy of the probe electrons has to be reduced such that their speed in the specimen does not exceed the one of light. For Silicon the maximum beam energy is 13 keV. Earlier studies with 20 keV still show a faint signature of these relativistic energy losses in the EELS spectrum [3].

In the present study we reduced the beam energy to the above mentioned theoretical Čerenkov limit of Silicon, which is 13 keV. The transmission electron microscope (TEM), FEI TECNAI G20, and the EELS spectrometer (GATAN GIF 2001) were aligned for this low beam energy resulting in an energy resolution of 0.7 eV. The single crystalline silicon specimen was mechanically thinned and finally ion polished using a 200 eV Ar⁺ ion beam. Directly before transferring the sample into the TEM the surface oxide was removed by a HF-dip using 24% hydrofluoric acid. The final sample thickness of the investigated area was chosen to be 40 nm in order to reduce the influence of surface plasmons on the low loss spectrum. Figure 1 shows the Silicon low loss spectrum as recorded and the single scattering distribution (SSD). The relative sample thickness for such low energetic electrons is 1.14 mean free path lengths for inelastic scattering. For retrieving the SSD the matrix deconvolution [4] was employed. In order to be able to compare the recorded data with optical measurements, a very small collection semi angle of 0.15 mrad was chosen.

Due to the fact that there are no final states available in the band gap of a semiconductor (or insulator) valence EELS (VEELS) makes the band gap energy directly measurable as the first spectral onset in the SSD. We find in perfect agreement to the handbook of optical data of solids [5] a value of $3.24 \text{ eV} \pm 0.06 \text{ eV}$.

When performing KKA one is able to retrieve the optical constants, such as the refractive index or the complex dielectric function. The most critical step within the data analysis is the removal of the zero loss peak (ZLP). In order to work as accurate as possible, we recorded a ZLP in vacuum under exactly the same experimental conditions as for the Si VEELS spectrum. This ZLP is subsequently used for the plural scattering removal routine leading to the SSD. The resulting SSD is further used for further KKA. Figure 2 shows the experimentally retrieved refractive index in comparison with the one from [5]. The agreement between our experiment and the optical data is unparalleled. Earlier studies [2,3] suffered from Čerenkov losses and from surface oxidation, which leads to a shift of the surface plasmon. A shift of the surface plasmon consequently leads to an error in all results of KKA in the 5 – 15 eV energy region. We still see a faint difference between our result and the optical measurements close to the band gap energy which can be lead back to the comparably bad energy resolution of the TEM. We are not able to resolve the interband transitions precisely enough. Also the shape of plasmon at 16.7 eV seems to depend on the excitation process.

Concluding there is to say that we were able to retrieve the dielectric function of Silicon employing electron beam techniques with unparalleled accuracy. The determination of the direct gap succeeded the first time because we were able to suppress relativistic energy losses completely.

References

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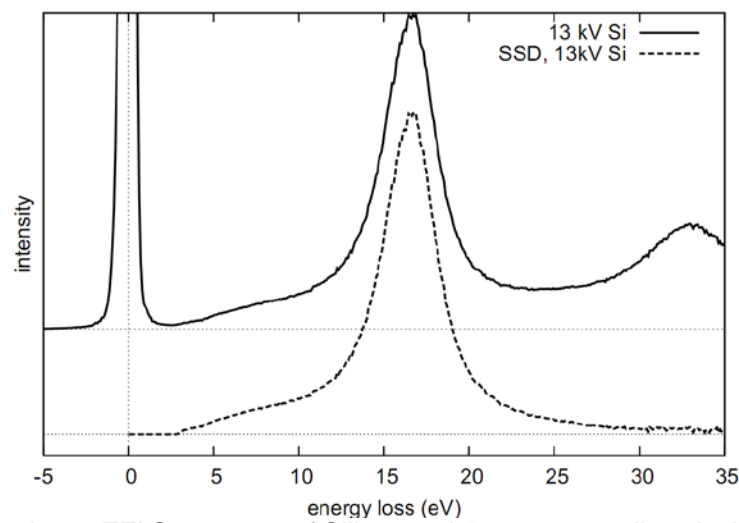


Figure 1. Raw 13keV valence EELS spectrum of Silicon and the corresponding single scattering distribution (SSD). The dotted lines give the zero values for the respective spectra.

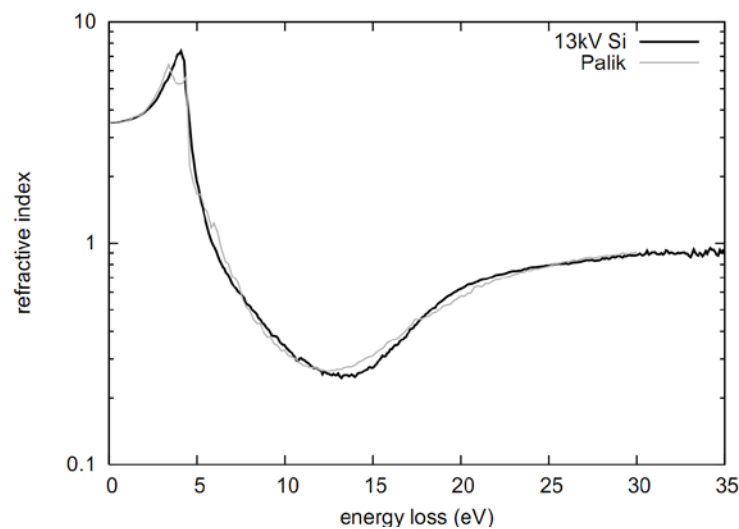


Figure 2. Refractive index obtained from the 13 keV experiment in comparison with optical data from the “Handbook of optical constants of solids III” [5].