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Issues Concerning Solid State Detectors for EXAFS

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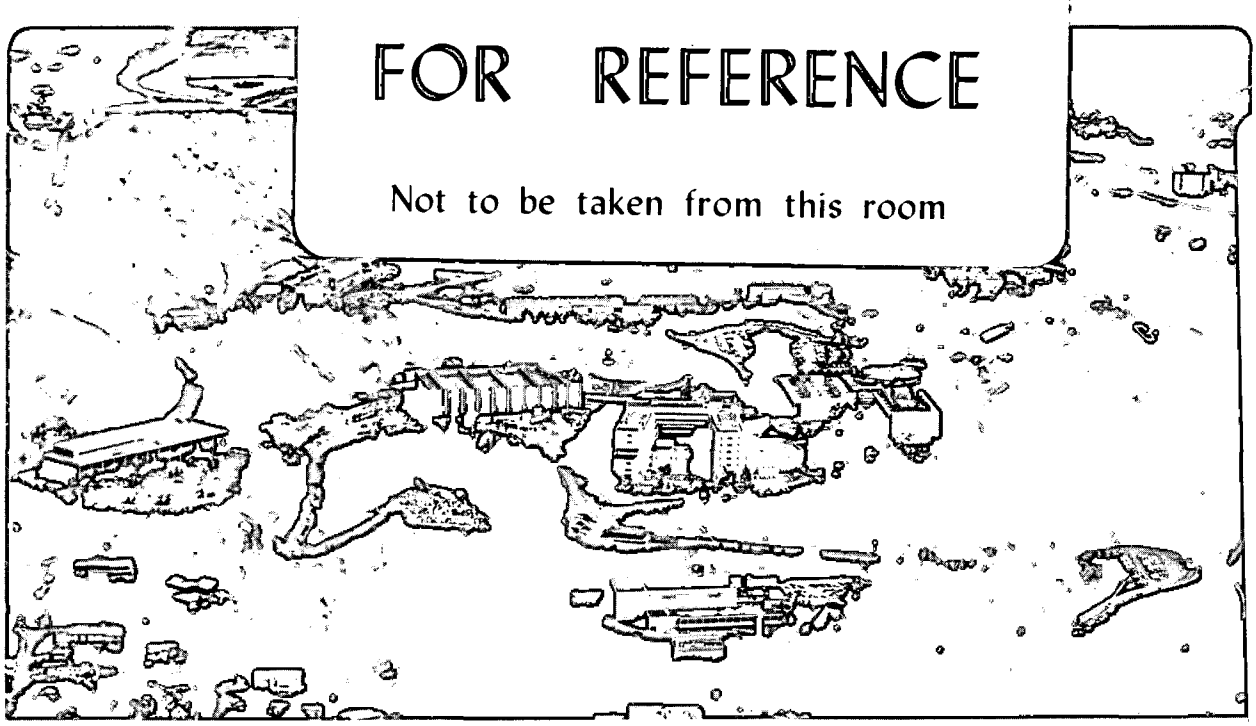

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Issues Concerning Solid State Detectors for EXAFS

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Introduction

Fluorescence extended x-ray absorption fine structure spectroscopy (EXAFS) is a commonly used technique in conjunction with x-ray synchrotron radiation for studying the local atomic structure of dilute elements in biological, geological and materials systems. Due to the nature of the EXAFS technique, and the difficulties associated with the detection of low energy x-rays, EXAFS has been used primarily in the energy range above 5 keV. However, there are a number of elements of interest with K- or L-absorption edges below 5 keV, which have not been easily accessible with existing EXAFS instrumentation. Figure 1 shows a representative x-ray spectrum which demonstrates several critical issues that must be addressed when considering optimum detector characteristics for low energy EXAFS measurements.(1) Referring to Figure 1, E_i is the energy of the incident radiation above the absorption edge of the element of interest, E_c is the energy of the incoherent scattered radiation and E_k is the energy of the fluorescent photopeak of the element of interest. The energy difference between E_c and E_k decreases as the energy of the fluoresced signal decreases, thus the detector resolution becomes increasingly critical as the atomic number of the element of interest decreases. For example, E_c - E_k for sulfur, with E_k equal to 2.3 keV, is only 150 eV. Although detectors are available which have adequate energy resolution for routine fluorescence applications, to fully exploit the intensity of synchrotron radiation, and also to maximize the signal-to-noise (S/N), the detectors must be run at high count rates which typically means shorter amplifier shaping timesand a concurrent degradation in energy resolution. Figure 1 also demonstrates the fact that EXAFS requires the measurement of a weak fluorescent photopeak on a large tailing background from the scattered photopeak. The attenuation of the fluoresced signal by the sample, and by the instrument and detector windows, will decrease the fluorescence intensity and decrease the S/N; the degree of tailing background from the scattered photopeak will strongly affect S/N. Recent work on Si(Li) and Ge detector windows and spectral backgrounds will be discussed relative to the aforementioned issues and relative to the development of a densely-packed Si(Li) array for EXAFS measurements.

Detector Windows

Regions of incomplete charge collection, or "dead" layers have been measured on Si(Li) and Ge detectors with a variety of contacts. The dead layer thickness is critically dependent on the type of contact and the contact processing technique. For example, Si(Li) detectors with Pd surface barrier contacts (Si(Li)/Pd) have thinner dead layers than those with Au contacts(2), and Si(Li)/Pd detectors have thinner dead layers than equivalent Ge/Pd detectors.(3) Dead layers in Si detectors with implanted contacts have been reported to be very thin(4), and likewise in Ge detectors with implanted contacts.(5) It is difficult to quantitatively compare dead layer thickness values obtained from different research studies using different measurement techniques, as the

interpretation of such measurements can vary, thus we will not attempt to do so here. Figure 2 shows the relative photopeak distortions which occur at low energies due to the dead layers in Si(Li)/Pd and Ge/Pd detectors, just above the Si K-edge and just above the Ge L-edge, respectively. The dead layers are estimated to be ~800 Å in the Si(Li)/Pd detector and ~2000 Å in the Ge/Pd detector. The thicker dead layers, in conjunction with larger absorption coefficients, result in larger photopeak distortions at low energies in the Ge detectors. The "shoulder" on the low energy side of the 1.77 keV photopeak from the Ge/Pd detector represents ~30% of the total counts in the photopeak. There is evidence to suggest that Ge detectors with amorphous-Ge contacts may offer significantly thinner dead layers than those with surface barrier contacts[6]; further investigation on these types of contacts is required.

Detector Spectral Backgrounds

Spectral backgrounds in Si(Li) and Ge detectors have been compared over a range of x-ray energies under equivalent measurement conditions. Figure 3 shows the relative photopeak/background ratios, as a function of incident photon energy, for representative Si(Li) and Ge detectors with Pd surface barrier entrance window contacts. The data were collected from the spectra produced by the excitation of characteristic x-rays from Ti, Cu, Se and Mo metal foils, using a Rh-anode x-ray tube.[3] Spectral backgrounds were higher in the Ge/Pd detectors, compared with the Si(Li)/Pd detectors, at equivalent incident photon energies; the spectral backgrounds were also higher in the Ge/Pd detectors when comparing the detectors more fairly at equivalent linear absorption coefficients. Figure 4 shows the relative spectral backgrounds from the fluorescence of a Ti foil, using Ge and Si(Li) detectors with different entrance window contacts.

Detector Arrays

Figure 5 shows schematics comparing the packing density of a commercially available Ge array used for EXAFS work and an LBL Si(Li) prototype which is being developed. The detector arrays are drawn to scale relative to each other, and are ~1 cm in diameter for the 13-element array and ~3 cm in diameter for the 5-segmented array. Calculations based on the scattering factors of a polarized synchrotron beam, as a function of detector solid angle and incident x-ray energy[7], show that the more densely packed 5-element array should yield a S/N on the order of three times higher than the more loosely packed 13-element array, despite the count rate advantages that a larger array affords. In addition, the use of Si, rather than Ge, as the detector material should further increase the S/N as the spectral backgrounds are lower in Si compared with Ge.

Discussion and Conclusions

Several characteristics of solid state detectors must be optimized for use in low energy EXAFS measurements. The detector entrance window, or "dead layer", must be as thin as possible to minimize the attenuation of the fluorescent signal. The detector spectral backgrounds must be minimized so that the tailing background on the low energy side of the scattered photopeak is as low as possible to maximize the S/N of the fluorescent photopeak. Based on our work, a thin Pd surface barrier contact on a Si(Li) detector

offers the thinnest detector dead layer and also the lowest spectral background for the Si(Li) and Ge detectors studied to date. To maximize the S/N, the detectors must be operated at as high a count rate as possible, without compromising detector energy resolution. High count rates can be achieved using multiple detector arrays; close packing of the detector elements can further increase the S/N by utilizing the "best" portion of the scattered polarized synchrotron beam.

Acknowledgment

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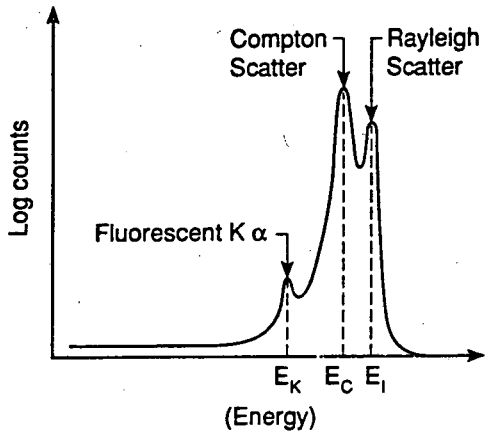

Fig. 1 Representative x-ray spectrum used in EXAFS measurements. E_i is the energy of the incident absorbed photon, E_c is the energy of the incoherent scattered photopeak and E_k is the energy of the fluoresced K-α x-ray from the element of interest. (Reprinted with permission from Ref. 1)

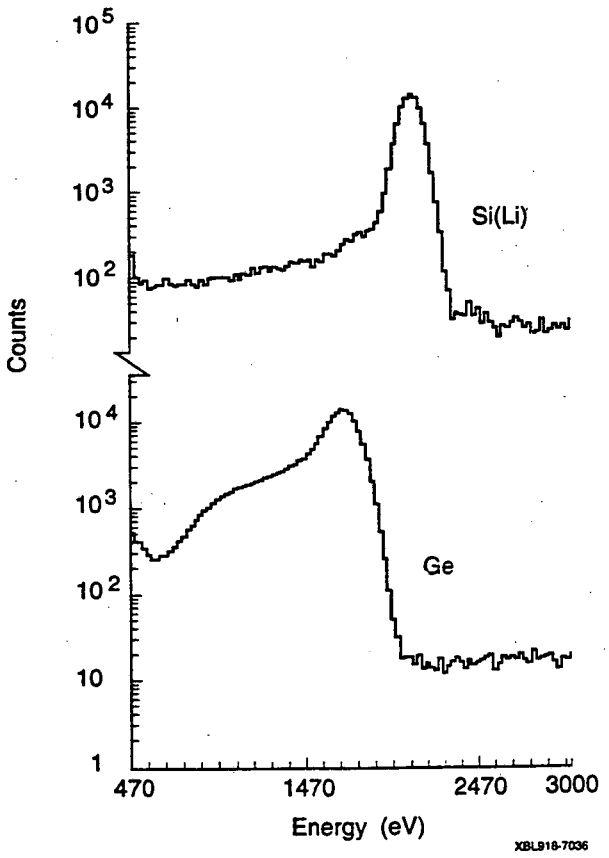

Fig. 2 (a) 2.0 keV spectrum from a Si(Li)/Pd detector, (b) 1.7 keV spectrum from a Ge/Pd detector.

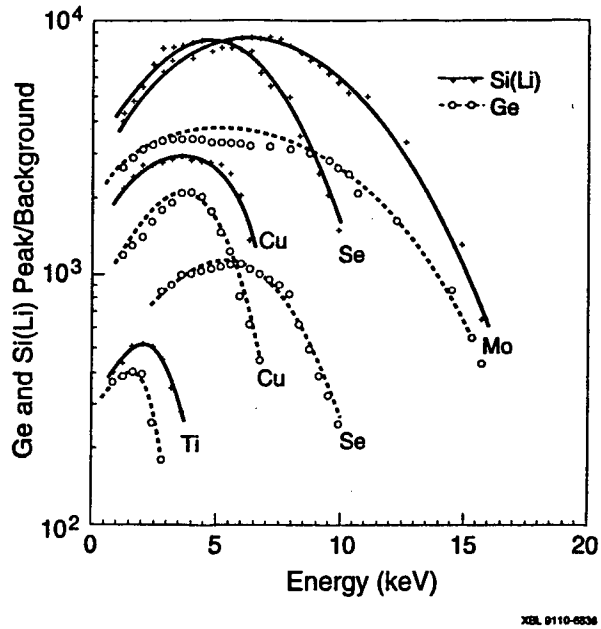

Fig. 3 Relative photopeak/background ratios for representative Si(Li)/Pd and Ge/Pd detectors, as a function of energy. The data were collected from the spectra generated from the excitation of characteristic x-rays from Ti, Cu, Se and Mo metal foils.[3]

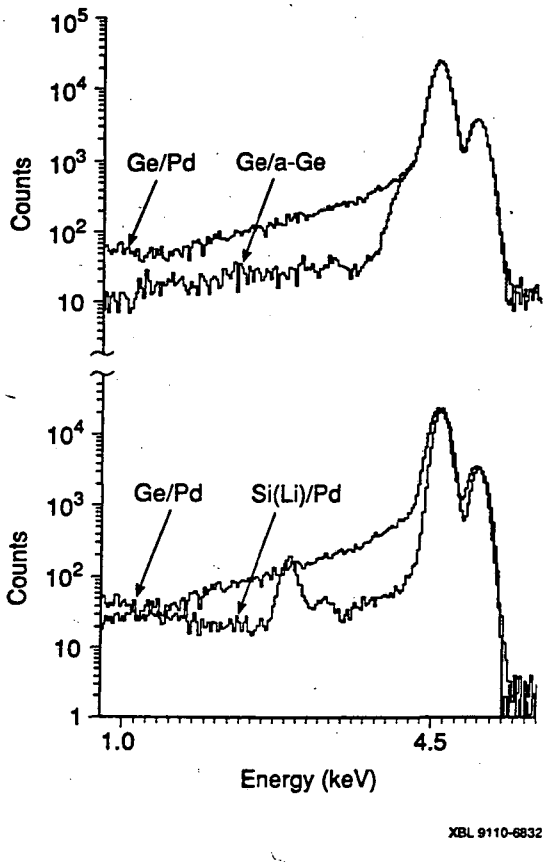

Fig. 4 Spectra generated from the excitation of characteristic x-rays from a pure Ti foil, using a Ge detector with a Pd contact, a Ge detector with an amorphous-Ge contact and a Si(Li) detector with a Pd contact.

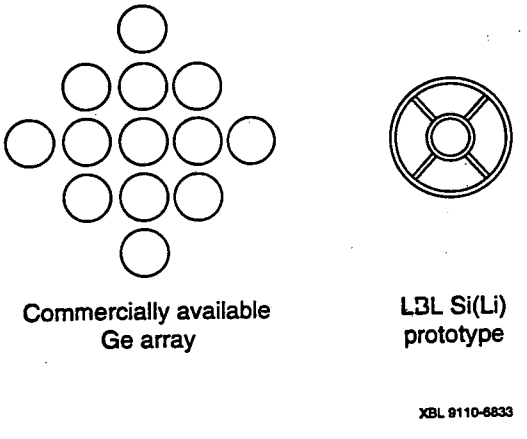

Fig. 5 Schematics comparing the packing density of a commercially available Ge detector array and an LBL Si(Li) prototype array. Ge detectors are ~1 cm in diameter; the Si(Li) array is ~3 cm in diameter.

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