Title
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Publication Date
1976-08-01

Presented at the International Conference
on Particle Induced X-Ray Emission and
Its Analytical Applications, University of
Lund, Lund, Sweden, August 23 - 26, 1976,
and to be published in Nuclear Instruments
and Methods

LBL-5368

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August 1976

Prepared for the U. S. Energy Research and
Development Administration under Contract W-7405-ENG-48

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SUMMARY

This paper presents some of the less recognized and potentially important parameters of the electronics and detectors used in X-ray fluorescence spectrometers. Detector factors include window (dead-layer) effects, time-dependent background and excess background. Noise parameters of field-effect transistors and time-variant pulse shaping are also discussed.

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1. INTRODUCTION

The past decade has witnessed the evolution of a new analytical technique which has required significant development of the method, of detectors and of the associated electronic systems. Notable steps include early detector developments and the use of cooled FETs as low-noise amplifiers(1-3), the invention and development of light feedback(4) and, somewhat later, pulsed-light feedback techniques(5), of low-background guard-ring detectors(6,7), and of the pulsed-excitation method(8). Some or all of these techniques are used in every present day energy-dispersive X-ray system. It has also been necessary to develop special X-ray tubes for use in photon-excited analysis systems(9).

It is a temptation to recite the history of these developments, but I prefer to take the opportunity to discuss some of the new or potential developments and some of the barriers to further progress. My viewpoint will reflect, in part, a strong bias toward the development of methods and techniques. However, it also draws from much experience in applying X-ray fluorescence to trace-element analysis starting in the early 1970's(10) and in particular to large-scale analysis of air particulates(11-13).

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* This work was done with support from the U. S. Energy Research and Development Administration and support from the U. S. Environmental Protection Agency .

2. DETECTORS

Without the development of semiconductor detectors, the energy-dispersive XRF method would not exist as an important analytical tool. The technology of silicon detectors has now reached a stable condition beset occasionally by the problem of obtaining high-quality silicon that permits high-detector voltage and low-charge trapping. From the view-point of the maker of detectors, a better understanding of the effect of material parameters and the availability of a steady source of good silicon would be the biggest steps that could now be made.

We are all intrigued, of course, by the hope that a higher band-gap material might become available thereby making room temperature operation of high-resolution detectors feasible. However, it is quite clear that high-resolution low-energy X-ray spectroscopy will be based on silicon for many years and that low-temperature operation will be necessary not only for the detector but also for the input amplifying FET.

I will now address a few detector problems that may not be well known and that can affect the accuracy of X-ray fluorescence analysis.

2.1 Window Effects

Much confusion exists about the so-called "dead layer" that exists at the entrance face of semiconductor detectors. This arises because various effects produced by the layer are important in different uses and because detectors are used at very different temperatures depending on the application. Thus, for example, many silicon detectors are used at or near room temperature for charged-particle spectroscopy. Here the most important effect of any "dead layer" is likely to be a downward shift in the position of peaks in the particle spectrum. An appropriate method of measuring the dead layer involves measuring the position of natural alpha-particle peaks for different angles of incidence of the particles on the detector surface. Using this method, very thin dead layers may be observed. For example, Elad, et al(14) observe dead layer thicknesses below 200 Å of silicon equivalent, most of which can be attributed to the metal layer which forms the surface barrier.

On the other hand, dead layers of much greater thickness have been reported by a number of authors(15-17). Eliminating the cases where poor processing (e.g. failure to totally drift lithium to the back in Li-drifted detectors) might be suspected, we find common reports of approximately 0.2 μm dead layers. Analysis of the observations shows that most were made in X-ray work although a few were in charged-particle experiments. However, all have the common feature that the detector was at low temperature (near 77°K). For the purpose of this conference it is important to recognize the existence of these thick dead layers and to register the fact that the thin windows measured at room temperature do not apply to X-ray spectroscopy.

We have recently been studying this problem and its effects and a full report authored by J. Llacer will be published shortly(18). Our study resulted from an attempt to use high-purity germanium detectors for low-energy X-ray spectroscopy--specifically for sulphur analysis. Since the average amount of energy required to produce a hole-electron pair is 20% smaller in germanium than in silicon one might hope for 20% better energy resolution in low-energy applications. Experiments quickly showed that a very large background existed extending from the sulphur peak down to zero energy and that this background was consistent with a layer approximately 0.3 to 0.4 μm thick at the entry surface of the detector from which only partial charge collection occurred. Further experiments showed that the layer was not due to poor processing and was present in all detectors.

Figure 1 shows the behavior of a high-purity germanium detector when irradiated by the K X-rays of several elements. These X-rays fall both above and below the L-absorption edge of germanium and the background is seen to jump drastically when the incident X-ray energy is just above the absorption edge. Taking all the background as being due to poor charge collection from a dead layer we find the results consistent with a "dead layer" thickness of 0.3 to 0.4 μm. In Fig. 2 the same effect is illustrated for a silicon detector where the jump in background occurs for elements whose X-rays are of somewhat higher energy than the silicon K-absorption edge. In this case the dead layer thickness would be judged to be 0.2 to 0.3 μm. The fraction of counts transferred to the background can be quite large--in the case of sulphur K X-rays, for example, the amount is as large as 50% in germanium and 16% in silicon.

This effect can be quite serious in producing incorrect results for light element analysis. The loss of counts in a peak is not in itself important since the system is calibrated with the same dead layer effect present. However, in a multielement sample, such as an air filter, where high levels of sulphur are usually present, the background tail from the sulphur peak must be taken into account in analyzing for elements whose X-rays are of energy lower than sulphur. Since the sulphur concentration varies greatly from sample to sample, the analysis program must "know" the shape of the background produced by sulphur and remove it in proportion to the amount of sulphur measured. The same behavior also applies to other elements. This problem, which can cause serious errors in determining light element concentrations, must be taken into account in analysis programs.

In view of the complications caused by the dead layer it is natural to seek the reason for it and for its apparently smaller value in room temperature charged-particle spectroscopy. The explanation appears to be that some of the electrons formed in the plasma (i.e. thermal electrons) at the photon interaction point diffuse into the surface (where they are lost) before their motion in the collecting electric field removes them from the region of the surface. This is not to be confused with escape of the original higher-energy photoelectrons which can escape from much deeper (~ 2 μm) in the detector. Detailed theoretical analysis requires application of Monte Carlo techniques with the competing energy-loss mechanisms taken into account at each electron collision, but a rough solution can be obtained by the simple diffusion analysis that follows:

Let: μ be the electron mobility

T be the temperature (°K)

V_s be the saturation velocity of electrons (assuming that the electric field is adequate to achieve this velocity)

k be Boltzman's constant

q be the electronic charge

Then: Average diffusion distance in time τ

$$= \sqrt{\frac{kT}{q}\mu\tau}$$

and: drift distance in the electric field $= V_s\tau$

Assuming that electrons might be lost if the diffusion distance exceeds the drift distance, it is reasonable to equate the dead layer thickness d to these two values:

$$d = V_s \tau = \sqrt{\frac{kT}{q} \mu\tau}$$

Eliminating τ we have:

$$d = \frac{kT}{q} \frac{\mu}{V_s} \qquad (1)$$

For germanium at 77°K, $\mu \simeq 4 \times 10^4 \mathrm{cm}^2 V_s^{-1-1}$, $V_s = 10^7 \mathrm{cm/s}$

$$\therefore \quad d = 0.28 \ \mu\mathrm{m}$$

This value, derived from such a crude model, is surprisingly close to the measured dead layer. Since both V_s and μ have about the same value for silicon at 77°K, roughly the same result is expected for silicon. Furthermore the reduction in mobility with increasing temperature, amounting to a factor ~ 40, is consistent with the big decrease in the dead layer at room temperature.

Therefore we conclude that the rather thick dead layer ~ 0.3 μm is probably due to this very basic physical process and represents a fundamental limit rather than being a consequence of manufacturing processes.

2.2 Time-Dependent Background

As discussed in our earlier papers, and by other authors(19), surface channels on detectors produce field distortions in the bulk which cause the charge due to events interacting in the "poor" regions to be partially collected in the surface layers. Figure 3 illustrates the behavior in a "grooved" type of detector with an n-type surface channel. In the measurement times used in spectrometry, the charge which flows into the surface is lost to the signal causing most of the degraded signals which constitute the background observed when using conventional detectors. The field distortions can exist over much of the volume of the small-area detectors used for high-resolution X-ray spectroscopy, so even the use of tight collimation of X-rays to the central region may not be completely effective in preventing some charge collection by surface layers. The guard-ring detector avoids this problem by defining the boundary of the sensitive region of the detector by internal electric field lines.

A second order (but important) effect of the collection of charge in surface layers is that the charge state of the surface may change during a short time when a detector is exposed to intense X-rays. The effect must always be in the direction tending to neutralize the surface states thereby reducing the field distortions and any resulting background. We have observed these time-dependent effects in detectors and find that the speed of the charge neutralization process and the slow decay back to normal background can be important in many X-ray fluorescence experiments and may affect the accuracy of results. Since the effect varies depending on the initial condition of the surface it is difficult to quantitate but should be evaluated for a particular detector and collimation system. The effect is absent in a properly manufactured guard-ring detector because the surface states are isolated from the sensitive detector volume.

2.3 Excess Background

In another paper to be presented at this meeting(20), I discuss the importance of detector-produced background in XRF analysis particularly in photon-excited systems where the scattered photons produce strong high-energy peaks in the spectrum. I also point out that the background level is substantially larger (approximately x 10) than might be expected on the basis of known physical effects. It is very important that the mechanism producing this background be understood. Since I have no good explanation to offer except (possibly) electron-channeling effects, I leave the mystery for future solution.

3. FIELD-EFFECT TRANSISTORS

The realization that field-effect transistors offered much better low-noise performance than vacuum tube amplifiers occurred about ten years ago and was followed quickly by their use at low-temperature to provide energy resolutions in the 300 eV range (FWHM). A few years later the value of removing the FET chip from its "noisy" package was realized and, by applying light feedback, we achieved the energy resolutions now common in silicon detector X-ray spectrometers. The last five years have been marked by little progress in low-noise FET development and by dependence of the whole XRF industry on selection of a few commercial types of FET with acceptance levels in the range of a few percent. At times the acceptance level has fallen essentially to zero and the whole growth of the XRF analysis method has been threatened.

This brief history should serve to point out that the present status of this critical item in XRF spectrometers is far from satisfactory. Apart from seeking a better understanding of noise parameters in present-day FETs to make possible more consistent performance, it is also desirable to aim toward development of new FETs with better performance. The user of XRF systems might well ask where improvements in FETs are required since the very best systems now achieve resolution adequate to resolve characteristic

X-rays from all but the lowest-Z elements. Furthermore, in the case of these elements, other serious experimental problems become dominant (sample and window absorption) while the resolution for the higher energy X-rays (~ 5 keV) is already very much affected by charge production statistics in the detector and improvements in electronic noise will have only marginal effect. However, the outstanding energy resolution in present day X-ray spectrometers is only achieved at the cost of long measurement times (~ 10 to 100 μs), a fact that seriously limits the counting rates at which the spectrometer can be operated. This reflects directly in the analysis time required to achieve a given sensitivity in XRF analysis.

Recent work by J. Llacer(20) has confirmed suspicions that the main source of the excess noise which causes the rejection of many FETs is the generation-recombination noise due to trapping impurities in the FET channel. This work has also developed a method to quickly analyze the traps and thereby to focus attention on important processing steps in FET manufacture. I now give a brief account of this work.

Most of the analytical work on FET noise in nuclear and X-ray spectrometers has dealt with the series (or delta) noise produced by fluctuations in the current in the FET channel and the parallel (or step) noise caused by fluctuations in currents (FET and detector leakage) or shunt resistance in the input circuit. These two terms in the noise are well understood and are represented by the relationship:

$$N = 2.35 \frac{\varepsilon}{q} \left[\left(q\, I_L + \frac{2\,kT}{R_P} \right) \langle N_S^2 \rangle + 2\,kT\, R_S\, C_{IN}^2 \langle N_\Delta^2 \rangle \right]^{\frac{1}{2}} \qquad (2)$$

where

N	is the noise line width (FWHM) expressed in eV
ε	is the mean energy required to produce a hole-electron pair in the detector
q	is the electronic charge
I_L	is the sum of the absolute values of all shunt leakage currents in the input circuit
k	is Botzmann's constant
T	is the temperature (°K)
R_P	is the parallel input circuit resistance
R_S	is the FET equivalent series noise resistance ($\simeq 1/g_m$ where g_m is the mutual conductance)
C_{IN}	is the total input capacitance (FET + detector + strays)
$<N_S^2>$ and $<N_\Delta^2>$	are the step and delta indicies(22) which are functions of the pulse shaping used in the system. These indicies vary as τ and $1/\tau$ respectively as the overall measurement time* τ is varied.

Note that C_{IN} appears in the second term because all noise is referred to the input and is expressed here in terms of equivalent energy absorbed in the detector.

It is well known that a third term must be added to this equation representing excess "1/f" type noise due to surface channels and other similar phenomena. This can loosely be regarded as step or delta noise coupled to the input circuit via a random distribution of integrators. This noise is best represented by a third term $A<N_{1/f}^2>$ added to the bracket in Eq. (2) where A has a value that depends on the device and $<N_{1/f}^2>$ is dependent on the pulse shaping network(23), but is independent of the measurement time τ.

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* The term "measurement time" is used here to represent the time scale of any pulse shaper used in the system. Typically τ might be interpreted as the peaking time of the pulse.

Figure 4 shows schematically the behavior of these three terms as a function of the measurement time used in the system. The magnitude of each component depends on the particular circuit parameters. In most X-ray spectrometers the minimum noise occurs for a measurement time in the range 10 to 100 μs.

This discussion has dealt with rather well-characterized FET parameters which, provide adequate understanding of the performance of FETs in room temperature applications. According to Eq. (2) a lower FET temperature should give less noise because T becomes smaller and the mutual conductance (g_m) increases making R_S smaller. In general, the noise does improve, but not as much as expected; also big variations are observed from one type of FET to another and between samples of a given type. Only the 2N4416 and its derivatives made by one manufacturer have proven useful in low-temperature low-capacity applications. The variability is caused by a fourth term which must be added to Eq. (2), the noise source being generation-recombination noise(24) caused by traps present in the gate depletion layer. The term which must be included in the bracket of Eq. (2) is $B\ C^2_{IN} <N^2_{GR}>$ where $<N^2_{GR}>$ is dependent on the pulse shaping network and varies with the measurement time τ as follows:

$$<N^2_{GR}> \propto 1/[2\ \tau_t/\tau + \tau/2\ \tau_t] \tag{3}$$

where τ_t is the characteristic generation time of the trapping level. The noise line width is therefore given by:

$$N = 2.35 \frac{\varepsilon}{q} \left[\left(q\ I_L + \frac{2\ kT}{R_P} \right) <N^2_S> + 2\ kT\ R_S\ C^2_{IN}\ <N^2_\Delta> + A<N_{1/f}>^2 + B\ C^2_{IN}\ <N^2_{GR}> \right]^{\frac{1}{2}} \tag{4}$$

If more than one trapping level is present, each must be represented by an additional term in the bracket with the value of $<N^2_{GR}>$ in a particular term depending on the level of the trap.

The behavior of the trapping-detrapping process causes most of the complications in the cooling behavior of FETs. At high temperatures the process is fast and fluctuations are at high frequencies and not effective in the measurement times used in our systems. At very low temperatures, on the other hand, any trapped charge is not released so no noise results. However, as the FET temperature is varied, a point occurs where the fluctuations due to a given trap are in the frequency range of the signal processing system. The temperature dependence is caused by the fact that the detrapping time τ_t in Eq. (3) of a single trap of energy E_t (where E_t is substantially less than half the band gap) varies as $\exp(qE_t/kT)$.

The noise behavior of a typical FET as a function of temperature is shown in Fig. 5. Normal detector systems operate at the temperature indicated in Fig. 5 at which minimum noise occurs. At very low temperatures (< 90°K) the rapid rise in noise is caused by deionization (i. e. freezout) of a small fraction of the main impurity (donor) atoms in the silicon. The noise is caused by fluctuation in the charge state of these atoms. The peak in the temperature range 90-130°K can be identified with an impurity causing a trapping level at ~ 0.2 eV in the band gap. The amount of noise in this peak varies from one FET to another, presumably due to a variation in the concentration of the accidental impurity. One or more further bumps are observed in the noise as the temperature rises toward room temperature. These can be identified with other impurities producing deeper trapping levels. It is obvious that absence of all these traps introduced by impurities would allow operation of the FET at slightly lower than the usual temperature with improved noise performance as shown dotted in Fig. 5. The best FETs are those with no bumps, but no FET we have tested totally lacks the low temperature bump at 90-120°K. This may be caused by oxygen-silicon complexes which would be impossible to avoid with standard FET manufacturing processes.

Two techniques are now employed by Llacer to measure these impurity levels. The first is to measure noise as a function of temperature in a conventional pulse-shaping system, but with the gate of the FET grounded. This eliminates the parallel input circuit noise and therefore clearly reveals the bumps. An example is shown in Fig. 6 where the main bump is clearly seen in the curve for the grounded gate mode but is only just visible in the normal mode. To accurately measure the energy level of a trap it is better to measure noise as a function of frequency at a fixed temperature. In a plot of noise vs. frequency the noise due to a trap appears as a "shoulder" in the noise plot (see Fig. 7). The frequency at which the shoulder occurs is dependent on temperature, as shown in Fig. 7, and its variation can be directly interpreted in terms of the trapping level. Using this method it is possible to identify the trap as a particular species (impurity or defect) and to study the effects of processing parameters on its concentration. The recent understanding of these mechanisms should stimulate progress toward having a reliable source of FETs of the type used in present-day X-ray spectrometers.

The longer-term problem of developing a better FET focusses on achieving better performance at short measurement times. As seen in Fig. 4 this requires reduction of the series (or delta) noise (the second term in

Eq. (2)) by producing FETs with better ratios of g_m/C where C is the FET input capacity. Using conventional FET designs this requires the use of smaller channel lengths and therefore better techniques of fabricating very thin lines on semiconductors. The Shottky barrier FET(25) offered promise in regard but these devices suffer from other problems such as high-gate leakage currents and "1/f" noise. Another logical step in FET development for low temperature applications is to use germanium rather than silicon. Donor or acceptor freezout would not then occur at 77°K and trapping levels are likely to be shallow enough that the noise bumps due to generation-recombination noise should be absent in the operating temperature range above 77°K. However, very different technologies are needed for processing germanium devices instead of silicon and these new processes would require development.

4. SIGNAL PROCESSING (ANALOGUE)

Signal processing involves both amplification and shaping of signals. A somewhat neglected aspect of amplification is the need for excellent gain stability and essentially zero drift in the baseline of signals. At first sight the gain and zero stability demands appear not to be too serious in X-ray systems since the fractional energy resolutions are rarely better than 1% while gain and zero stabilities are usually in the region of 0.05% of full scale in a well-designed system. However, spectral stripping procedures in a computer are very sensitive to slight peak shifts and significant residuals can appear after subtraction of a spectral peak if it has moved from its reference position by more than 0.01% of full scale, (i.e. by 0.1

channel in a 1000 channel spectrum). These residuals can seriously affect the accuracy of determination of a low-concentration element whose characteristic line is very close to that of a common element (e. g. Mn in the presence of Fe). These effects demand excellent stability of the whole system and make it necessary for computer program to correct for gain and zero shifts by using fiducial peaks in a spectrum as reference marks.

The problem of optimum pulse shaping has received as much attention as any in nuclear instrumentation over the past three decades. Practically all X-ray spectrometers now in use employ the pseudo-Gaussian pulse shaper, originally proposed by Fairstein(26), using active integrators equivalent to as many as seven RC integrators. It is well known that a cusp-shaped pulse can give slightly better noise resolution but it is sensitive to variations in collection time in the detector and is not very suitable for later processing such as stretching.

The subject of time-variant pulse shapers has received much attention in recent years(27) and specific applications of the gated integrator(28) have been published. The gated integrator, fed by a Gaussian shaper, may soon be applied to X-ray systems since its noise resolution can be somewhat better for a given measurement time than the simple Gaussian shaper(22). Until now little use has been made of time variant differentiation because a noise penalty normally results from its use. A new application of this technique permits fast processing of the dominant large-amplitude backscatter pulses seen in photon-excited fluorescence spectrometers while normal long processing times are employed for the smaller pulses of more interest. This scheme permits much higher total counting rates since little time is wasted in processing the frequent backscatter pulses. This idea has been discussed for some time but has now been applied by Desi(29). Figure 8 shows his scheme. A delay is inserted in the main signal channel and a parallel fast channel is used to switch the main differentiator to a small value when the fast discriminator senses a large pulse. In Desi's system integration times in the main shaper are unchanged so the effect of the short differentiation is that large input pulses become very small (but long) pulses after passage through the normal signal channel. A better scheme would use a gated integrator switched to store no charge at the same time as the differentiator is switched to its short time constant. In some X-ray analysis programs, the size of the backscatter peaks is used for background normalization and appearance of at least a known fraction of the backscatter events in the output spectrum is essential. This can easily be accomplished by inhibiting the fast channel to permit normal processing of a selected fraction of the large pulses.

Another important development is the integrated system designed by Kandiah, et al(30) for processing pulses in X-ray spectrometers. In this system, pulsed-light feedback is used to reset the preamplifier input on each pulse, the entire signal processing chain is dc-coupled to avoid high rate problems and time-variant (both a switched differentiator and a gated integrator) shaping is employed. The unit described by Kandiah et al, shown in block form in Fig. 9, is designed as an integral processing package with convenient operator controls. This idea may well become the basis for the design of future X-ray spectrometers. It readily lends itself to implementing schemes like that of Desi whereby pulses in a selected amplitude range are processed differently from those of other amplitudes.

5. CONCLUSION

The design of detectors and electronics for X-ray fluorescence spectrometers has reached a fairly stable state and apart from some of the details discussed here significant changes cannot be expected in the next few years. Perhaps the most important single item demanding further work is the FET where new developments are desirable but, even more important, a better understanding and control of the parameters of the 2N4416 type of FET must be achieved. A better understanding of the factors influencing detector background may also be important in improving the sensitivity of the X-ray fluorescence method.

6. ACKNOWLEDGEMENTS

The comments in this paper owe much to discussions with D. Landis, N. Madden, J. Llacer, G. Haller, J. Walton, J. Jaklevic and B. Loo.

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FIGURE CAPTIONS

Fig. 1. Spectra obtained for monoenergetic X-rays incident on a germanium detector. The X-ray energies in the lower half of the figure lie above the Ge L-absorption edge while the one in the upper half is below the edge. The sudden increase in background as the X-ray energy changes from 1.19 keV to 1.25 keV is seen; this corresponds to a Ge dead layer approximately 0.4 μm thick.

Fig. 2. Similar to Fig. 1 but for a silicon detector. While the background level is considerably lower than in the Ge detector a similar step in background occurs as the X-ray energy exceeds the K absorption edge of silicon. The background is consistent with a silicon dead layer ~ 0.3 μm.

Fig. 3. Shows the field and potential distribution present in a typical silicon detector 5 mm diameter and 4 mm thick with a rather heavily n-type surface. Charge produced in the shaded regions is partially collected in the surface layer resulting in degraded signals.

Fig. 4 Typical behavior of the series, parallel and "1/f" noise terms in a detector-FET combination shown as a function of the measurement time. The series component increases as the input circuit capacity increases.

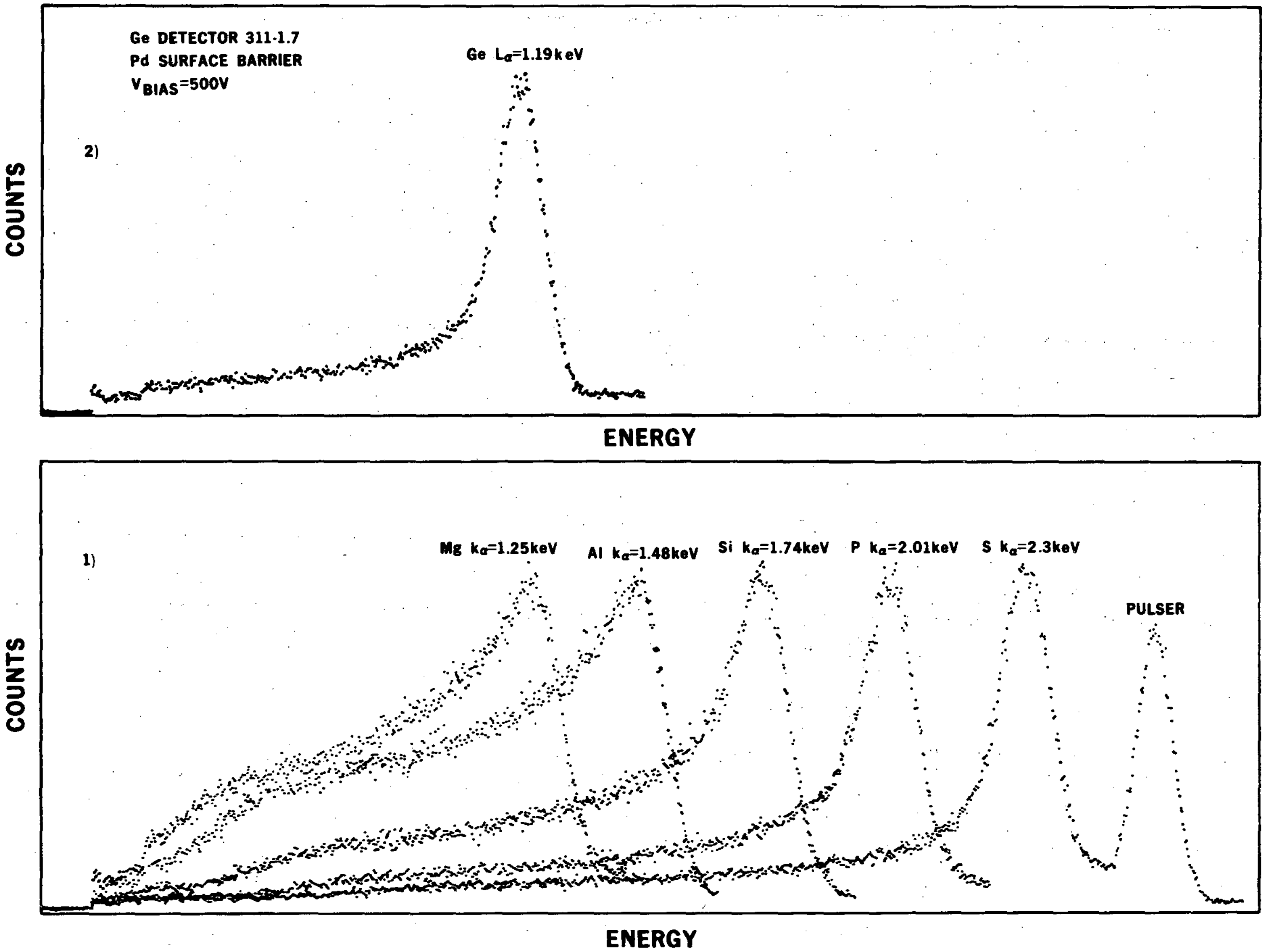
Fig. 5. Typical behavior of the noise in an FET-detector system as a function of temperature. The dotted line shows the performance that might be achieved if generation-recombination noise produced by traps were not present. These curves are given for the FET (2N4416) in its normal header.

Fig. 6. Illustrating the advantage of measuring FETs in the grounded gate configuration to determine the generation-recombination noise term. The removal of parallel noise terms makes the effects of traps much more obvious.

Fig. 7. A frequency domain plot of FET noise showing the effect of a single trapping level. The shoulder observed in these curves is produced by G-R noise from the trap and is temperature dependent as expected.

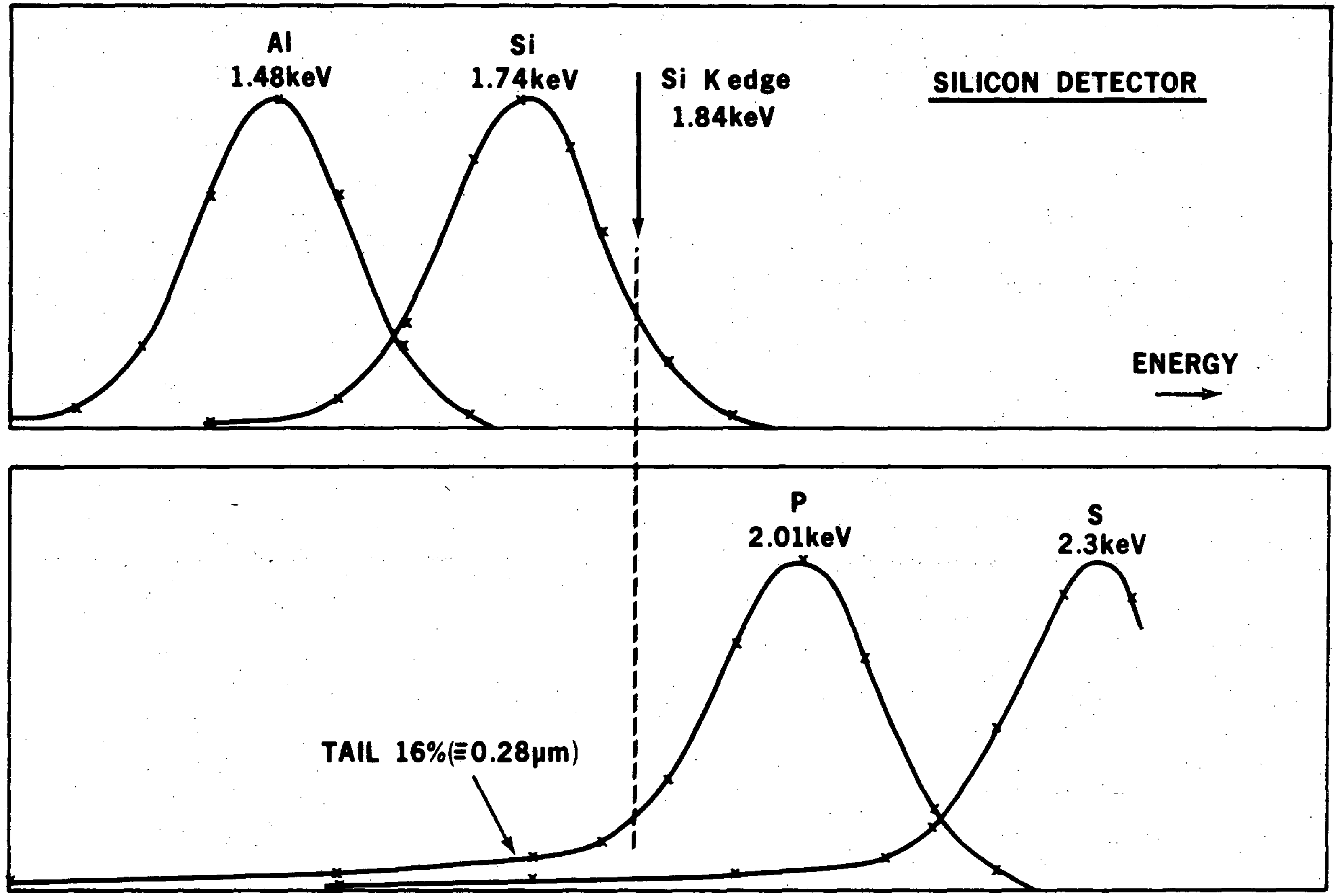
Fig. 8. Block diagram of the system used by Desi (Ref. 29) to reduce the processing time for large pulses in an X-ray system.

Fig. 9. Block diagram of the "integrated" processor for X-ray spectrometry described by Kandiah (Ref. 30).



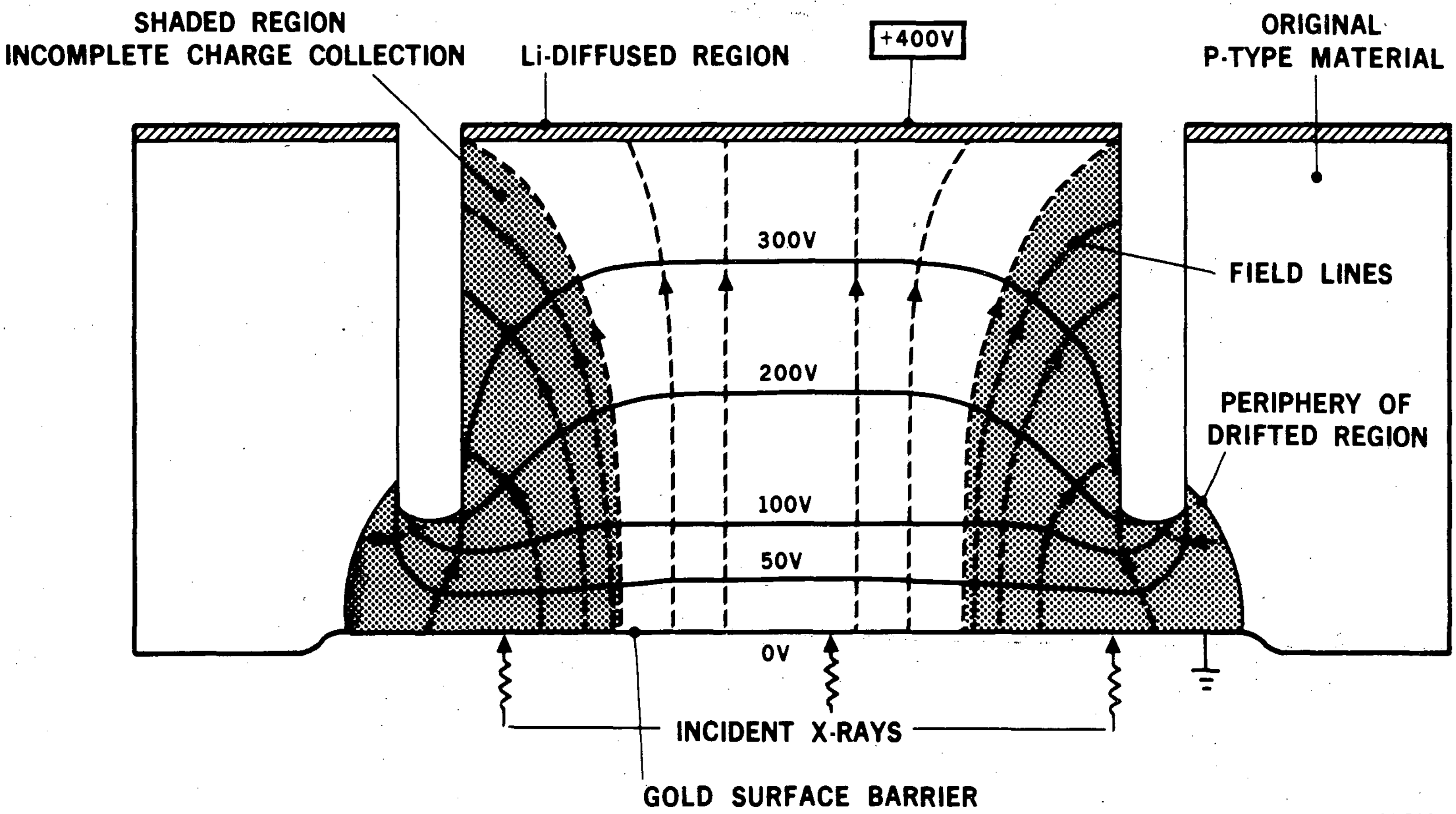
XBL 764-1610

Fig. 1



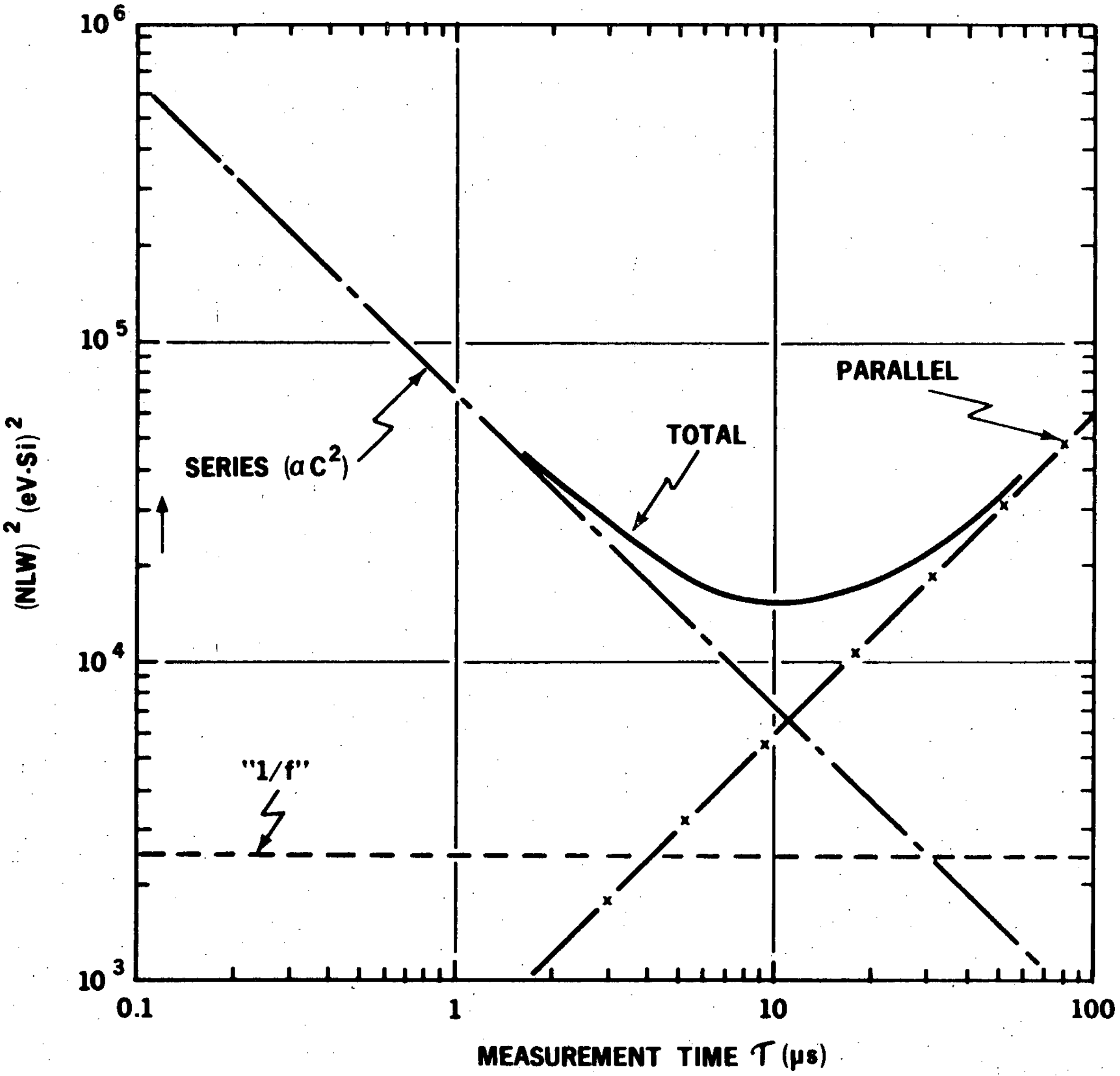
XBL 767-8887

Fig. 2



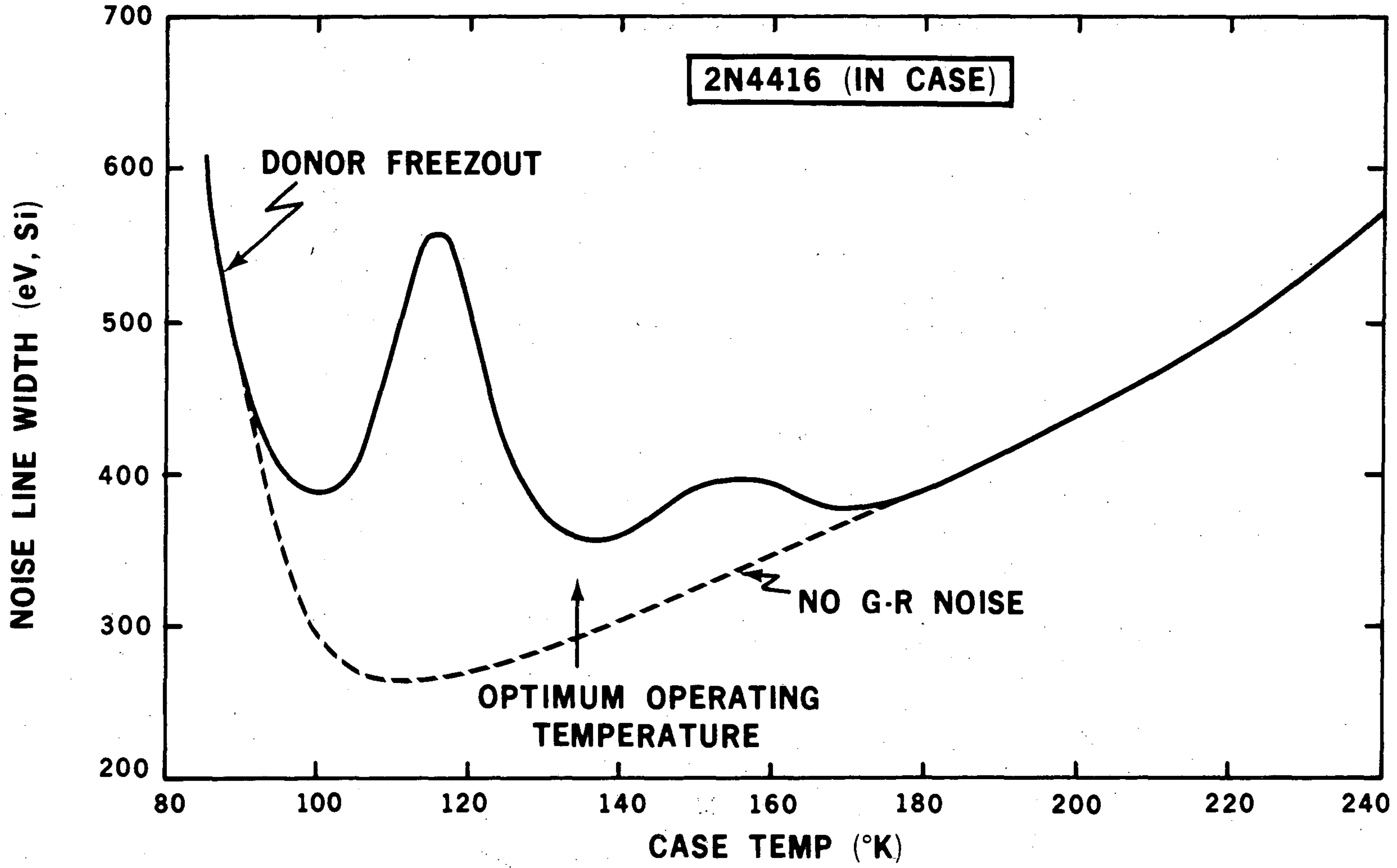
XBL 767-8891

Fig. 3



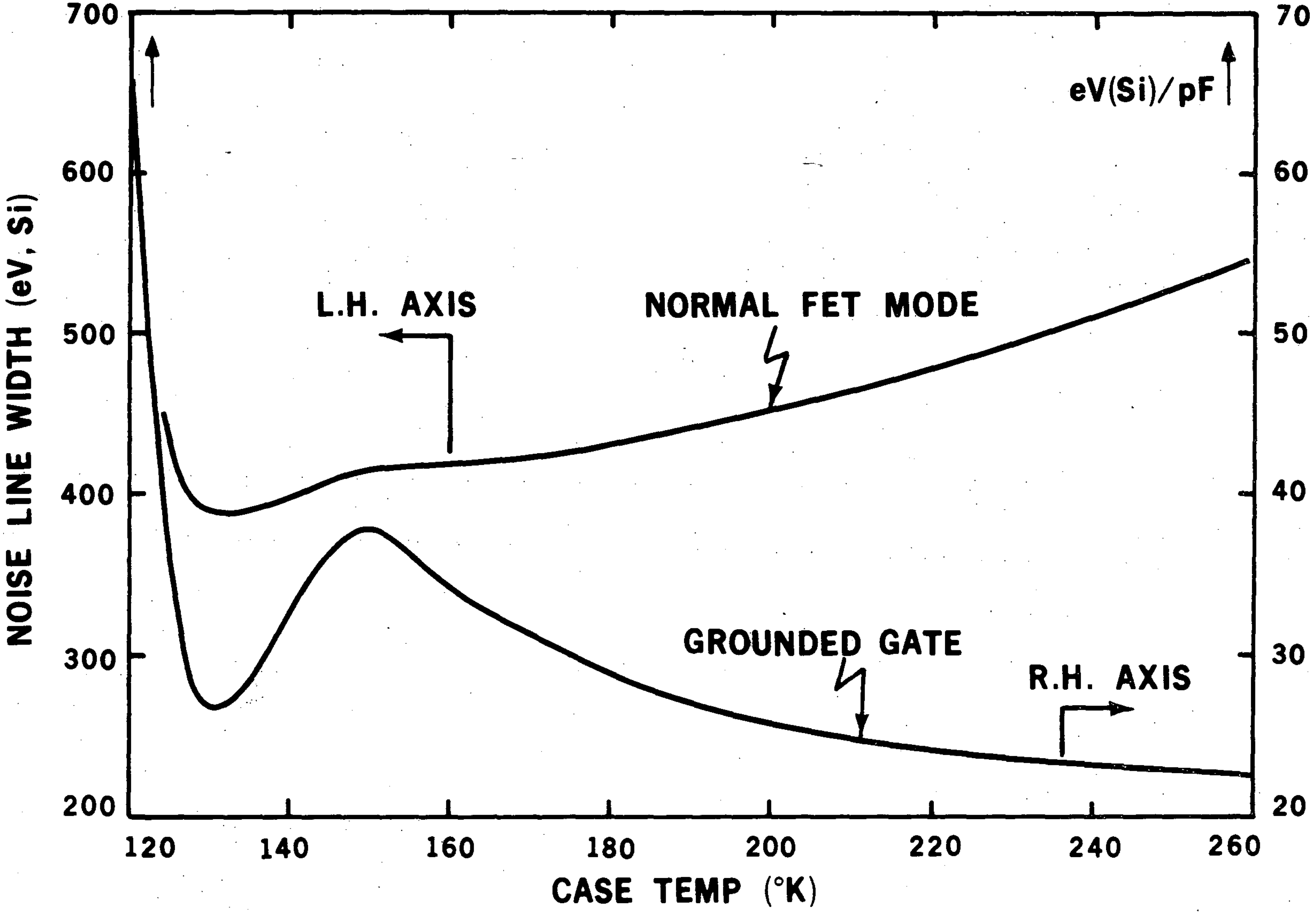
XBL 767-8893

Fig. 4



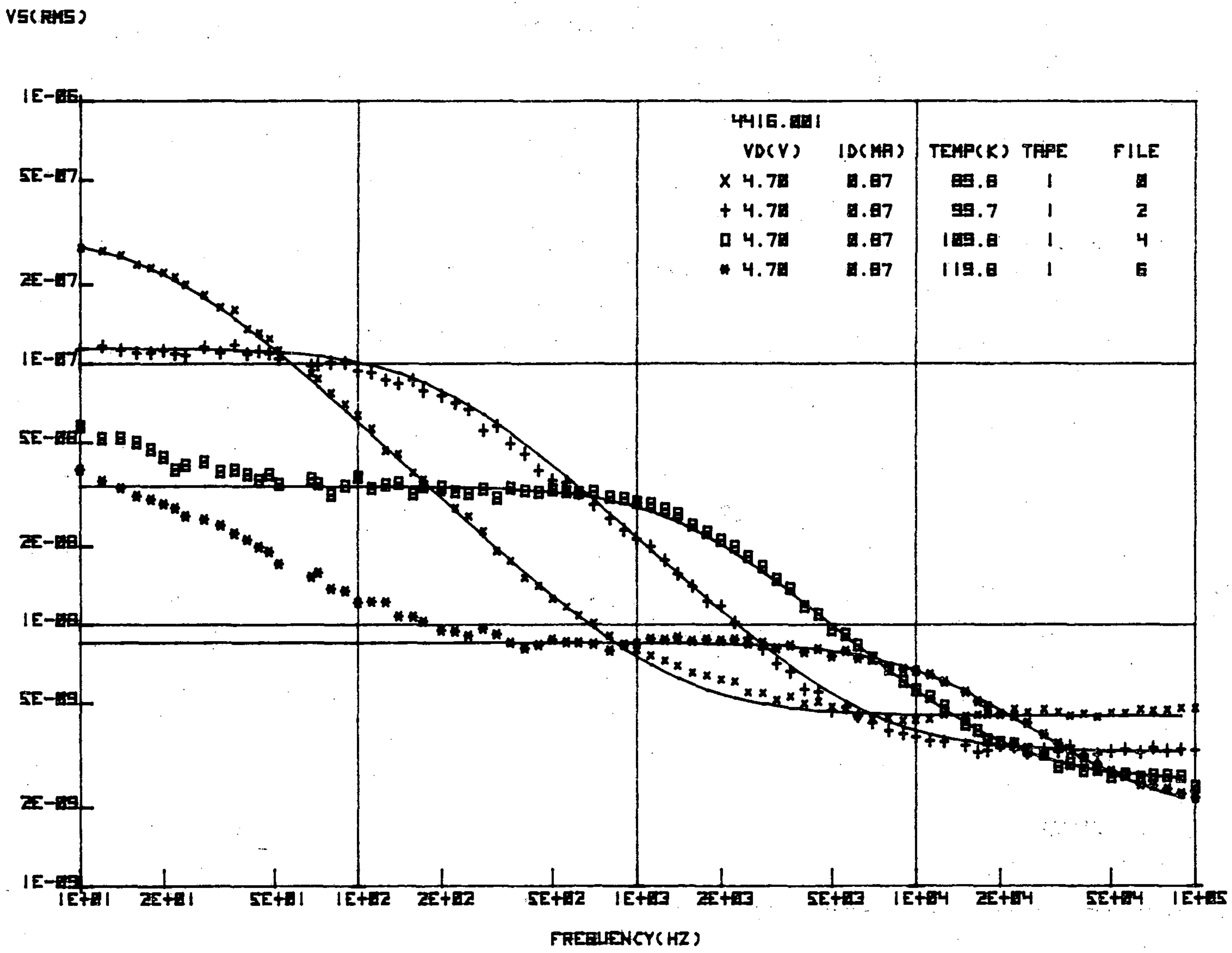
XBL 767-8889

Fig. 5



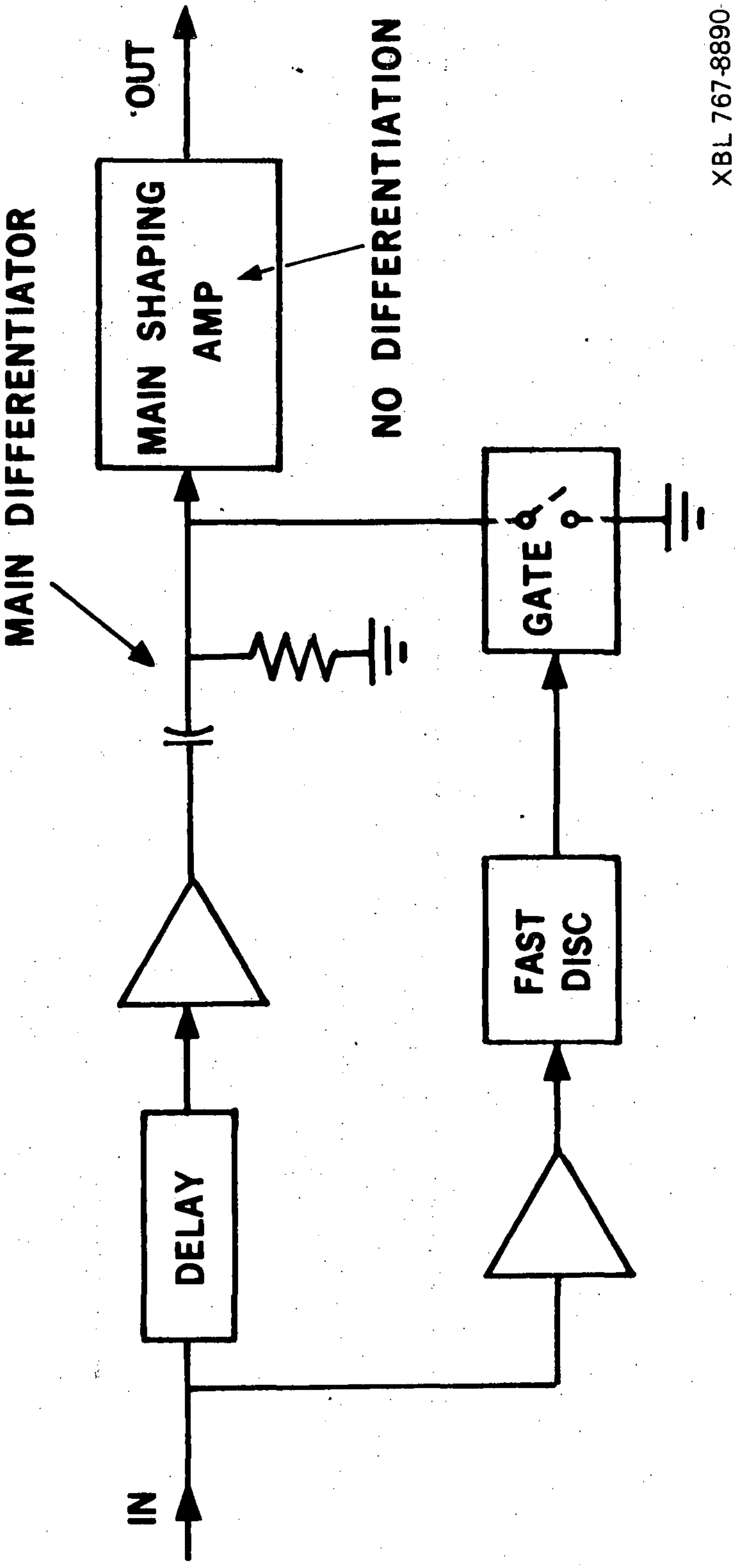
XBL 767-8888

Fig. 6



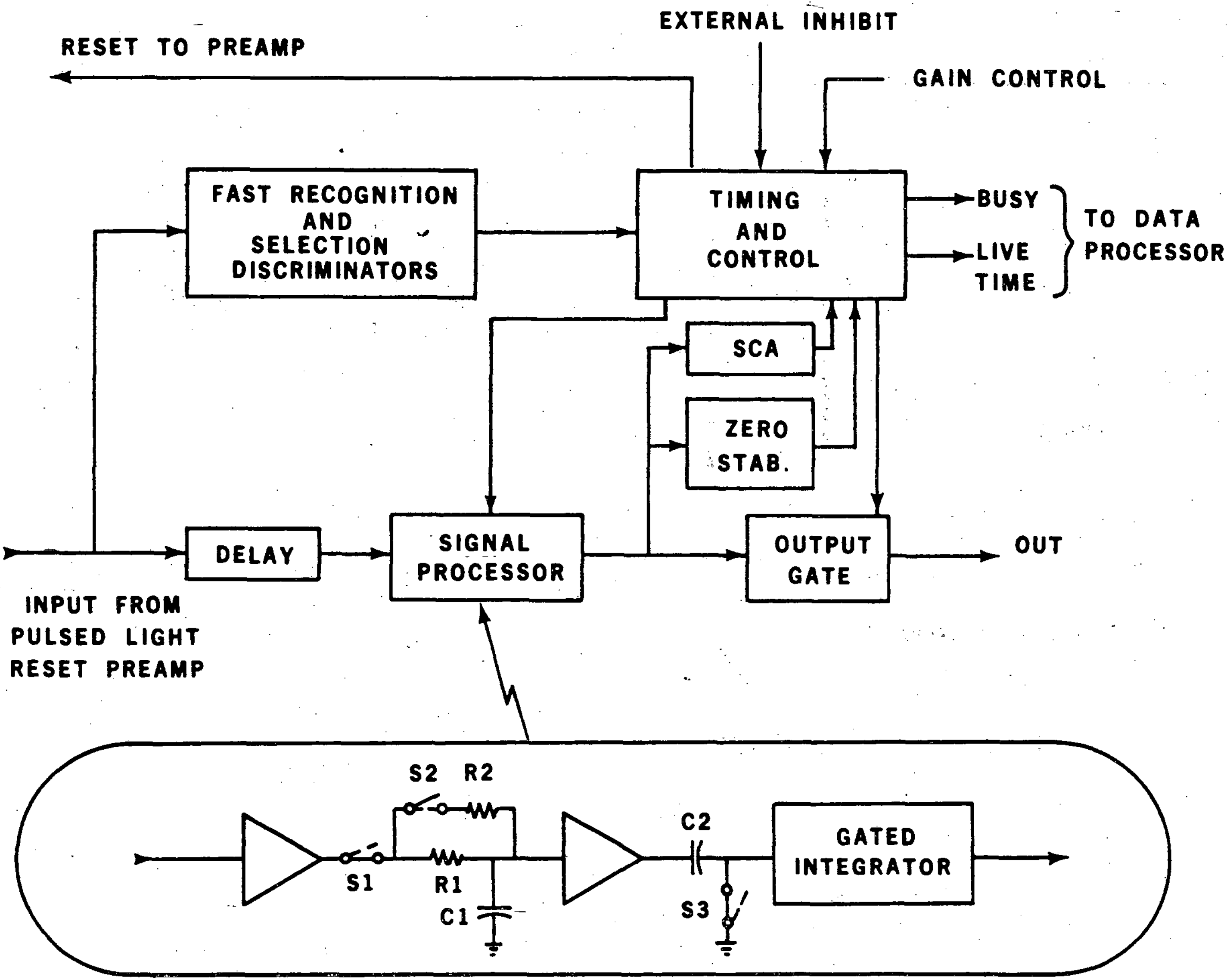
XBL 764-1608

Fig. 7



XBL 767-8890

Fig. 8



XBL 767-8892

Fig. 9

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