

X-ray Detectors III

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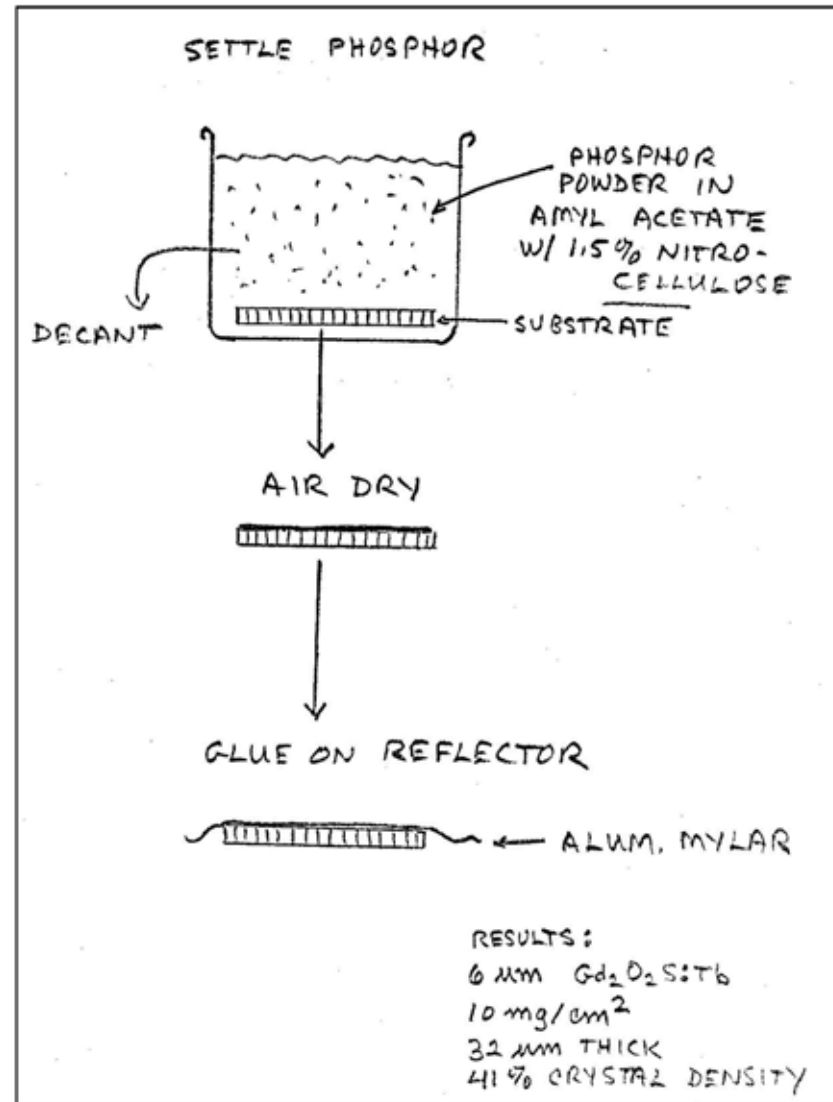
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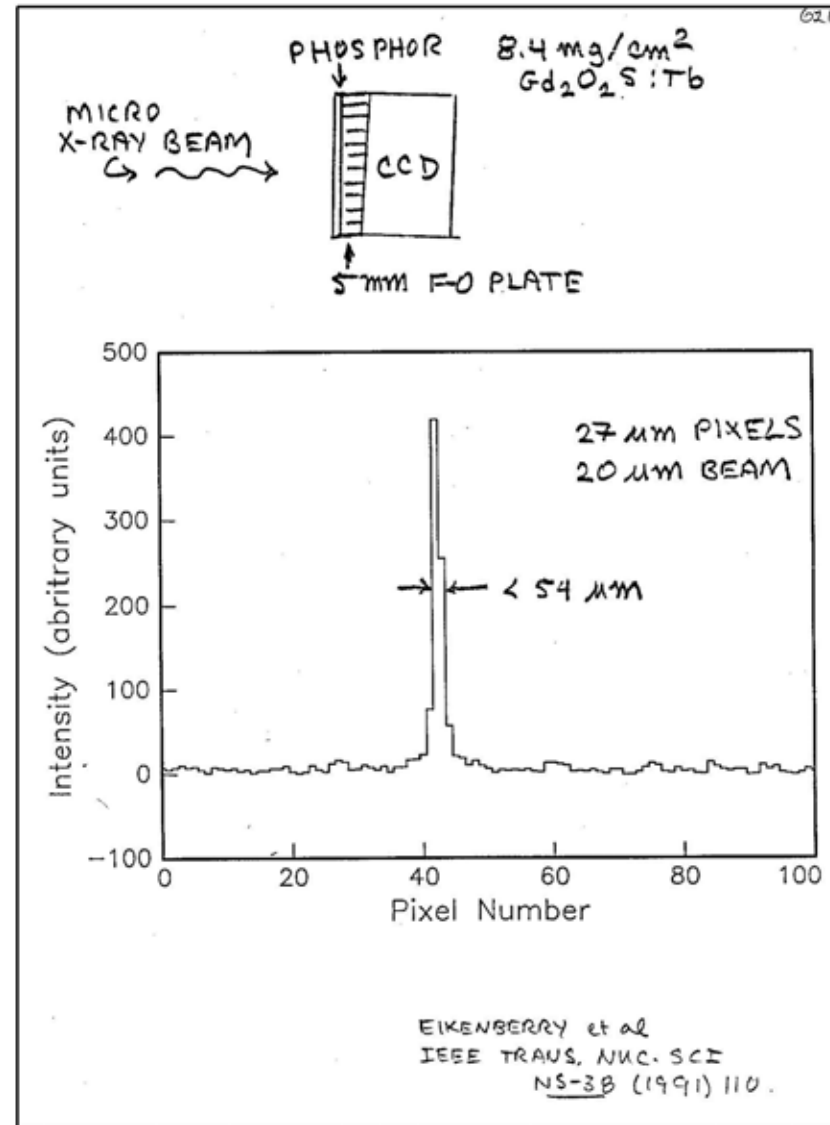
Settled Phosphors

Surprisingly good quality phosphor screens can be made by settling 1 - 10 micron grains of phosphor. This is important because some of the best x-ray phosphors are only available as powders. See Gruner et al, *Proc SPIE*, 2009 (1993) 98.



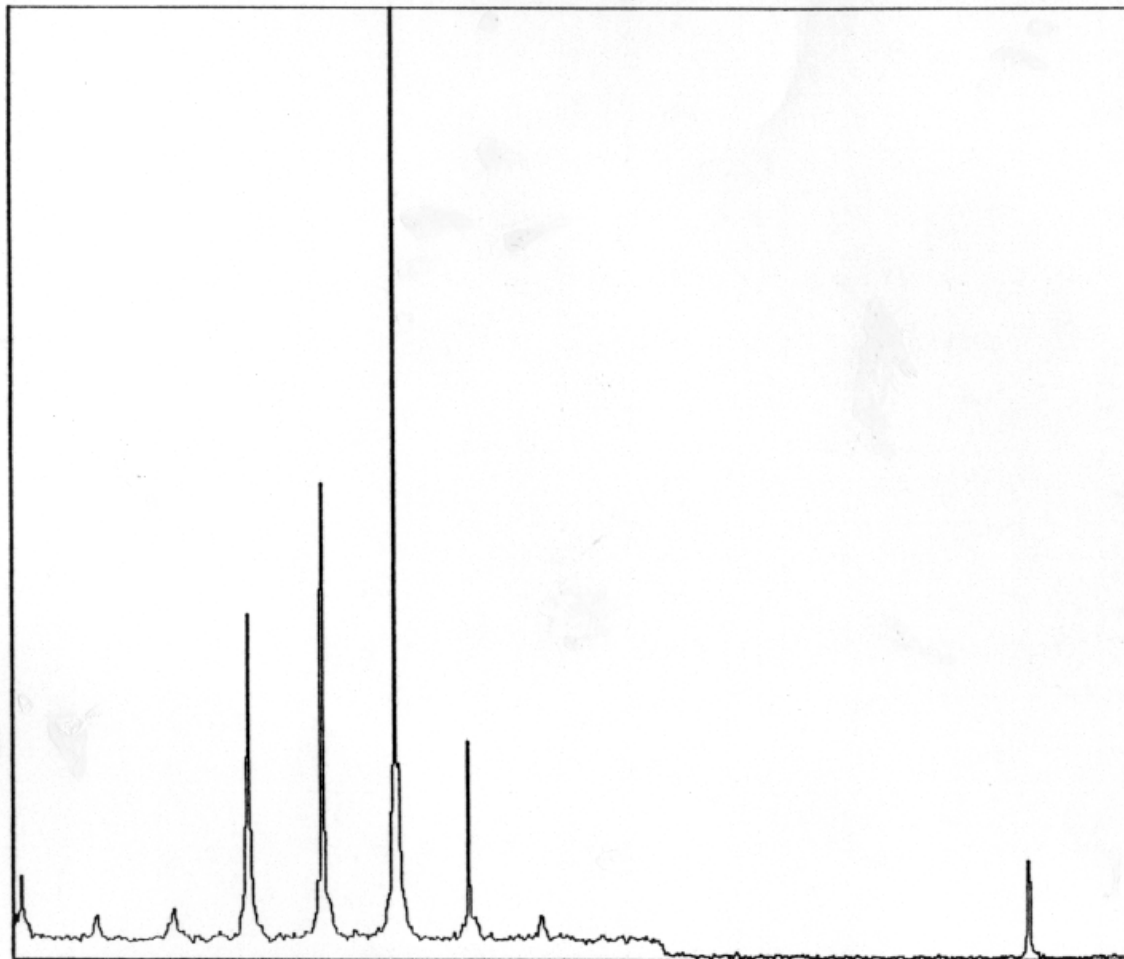
Settled Phosphors

The PSF of a properly settled phosphor powder screen is remarkably good. These screens scatter light strongly and one might intuitively expect a PSF with a wide base. In fact, this is not observed.



X-ray Detectors II

GaAs Laue pattern. Note that many peaks are a single 27 micron pixel wide.

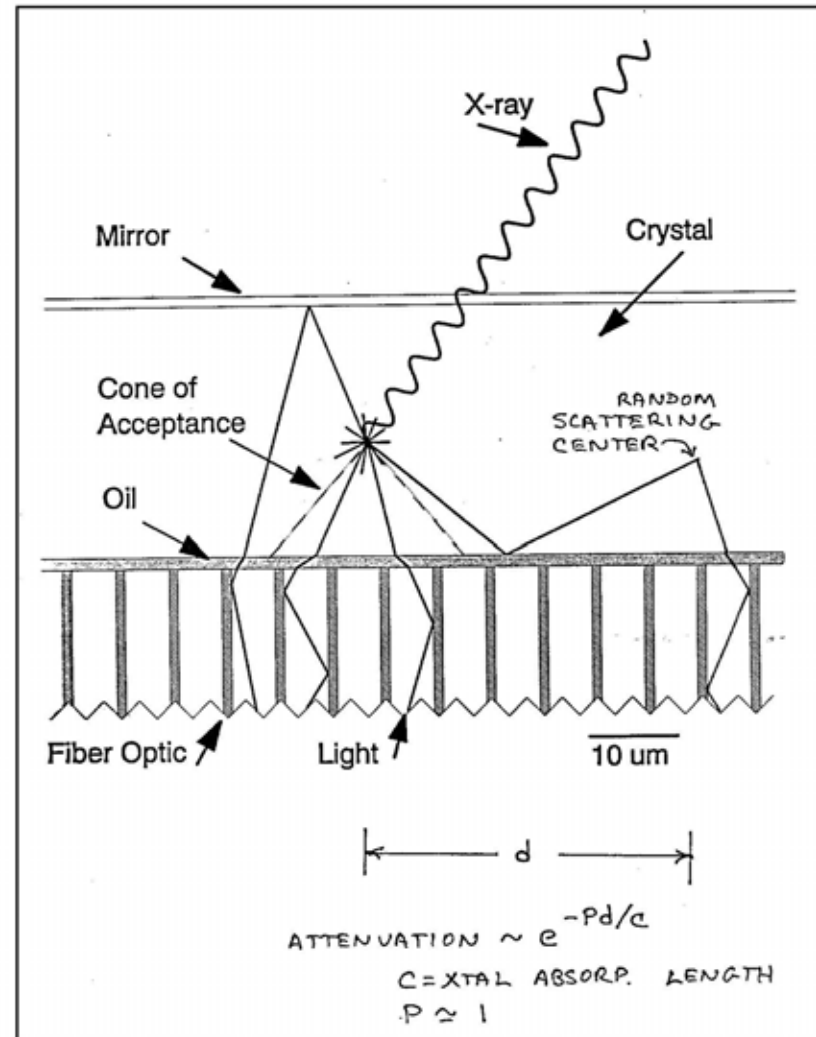


Eikenberry et al, in *Photoelectronic Image Devices*, 1991, E.L. Morgan, ed. (Inst. Phys Conf. Series No 121, Inst of Phys, Bristol, UK, pp 273-280)



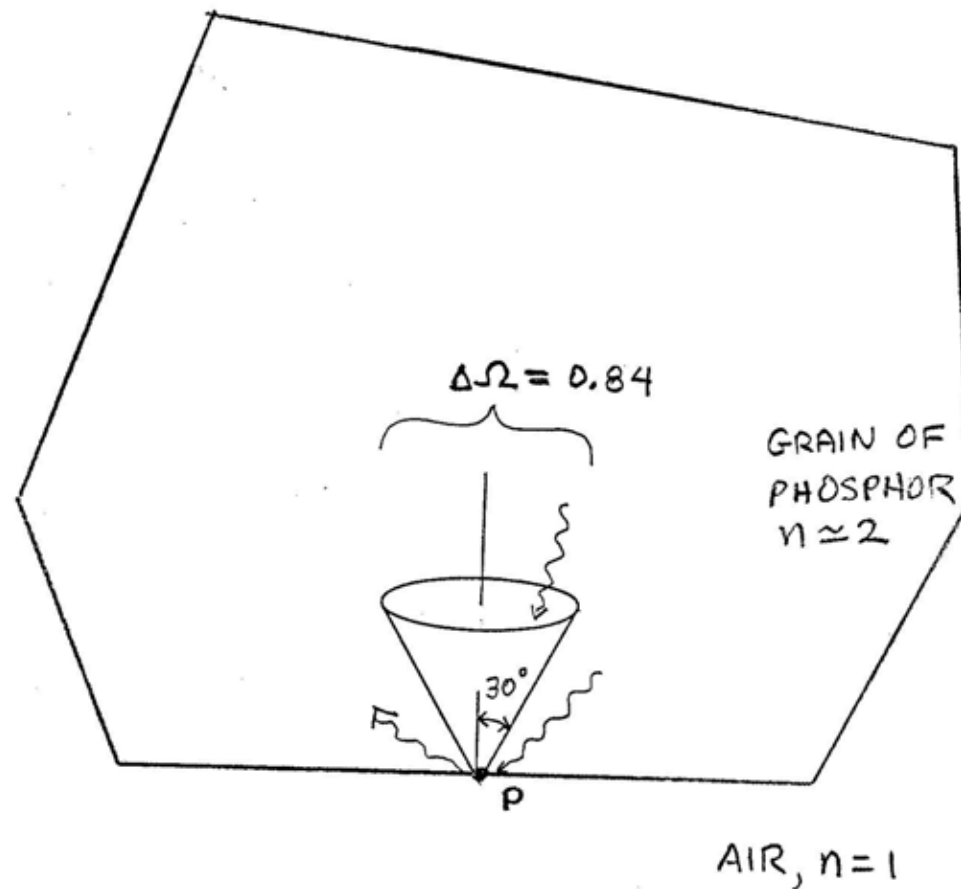
Settled Phosphors

The key to understanding settled screens is the observation that their PSF gets *worse* if the screen is infiltrated with an index matching fluid. The ideal is to fabricate the screen so that the light photons execute a random walk through the screen, with a step size equal to the mean phosphor grain size, e.g., ~ 6 microns (Gruner et al, *Proc SPIE*, 2009 (1993) 98). First, consider a single crystal screen:



Settled Phosphors

For high index powders, the probability of light escape into air is very low:

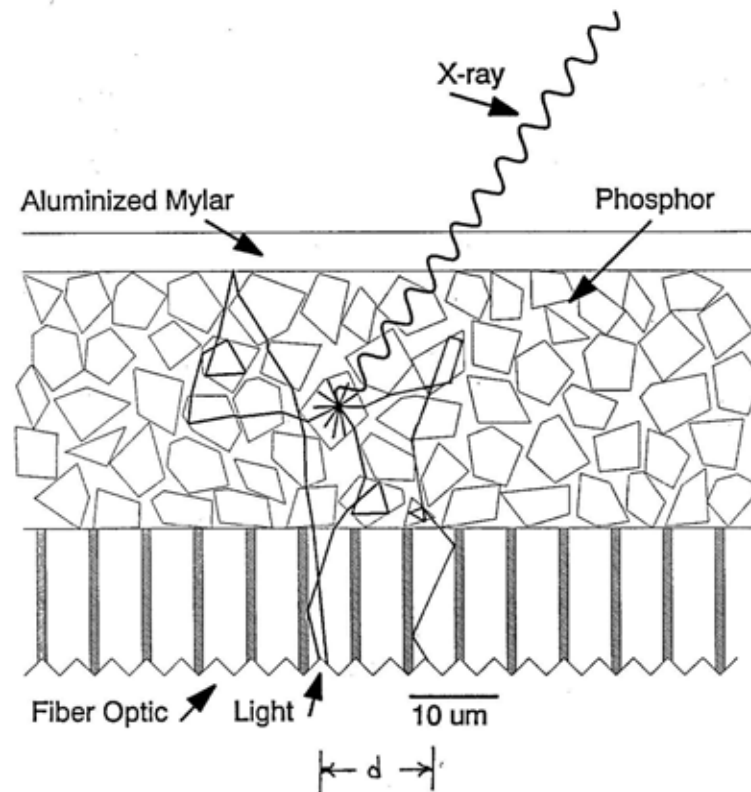


PROBABILITY FOR ESCAPE
AT POINT P = $\frac{\Delta\Omega \text{ (STERAD)}}{2\pi \text{ (STERAD)}} = 0.13 \approx \frac{1}{7.5}$



Settled Phosphors

So light takes a random walk through powder screens with low index gaps between the grains (From Gruner et al, *Proc SPIE*, 2009 (1993) 98):



$$d \approx S\sqrt{N}$$

$$d_{TOT} = SN$$

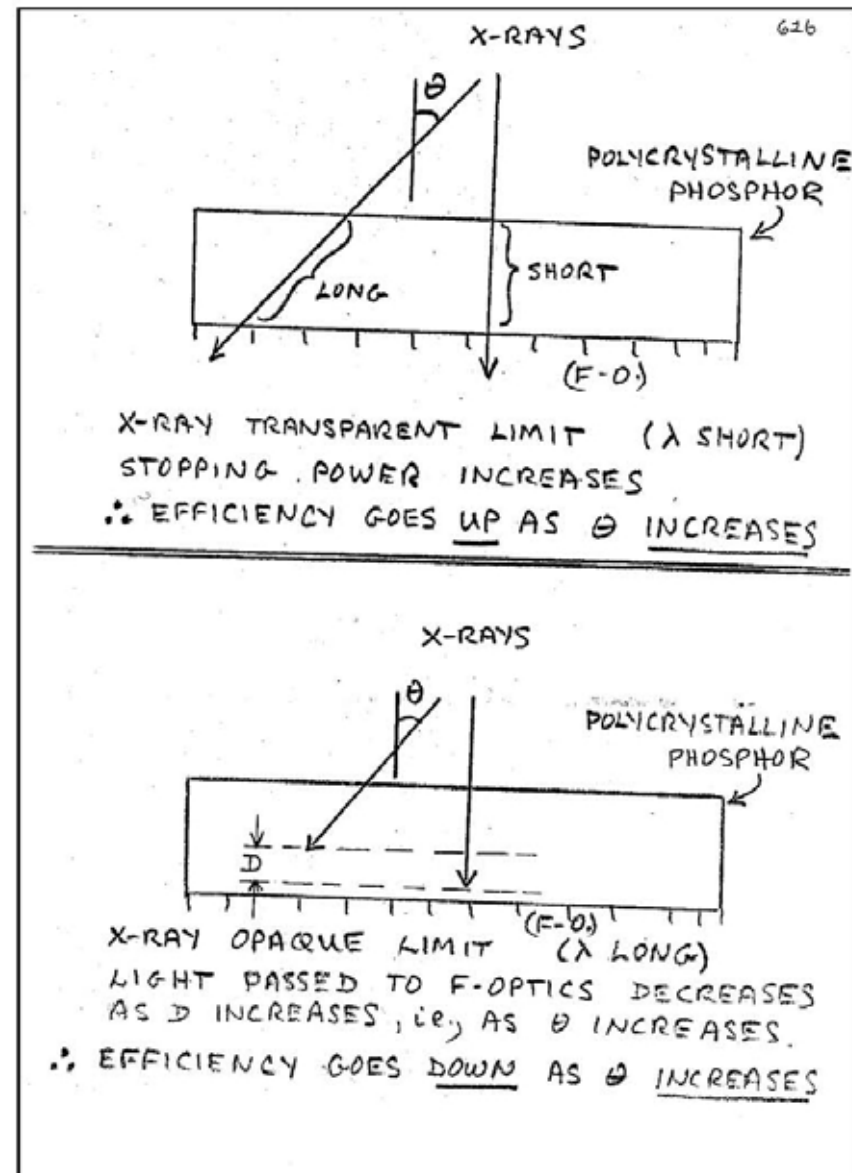
$$ATTENUATION \sim e^{-\frac{d_{TOT}}{c}} = e^{-\frac{d^2}{cS}}$$

$S = \text{STEP SIZE}$
 $N = \# \text{ STEPS}$



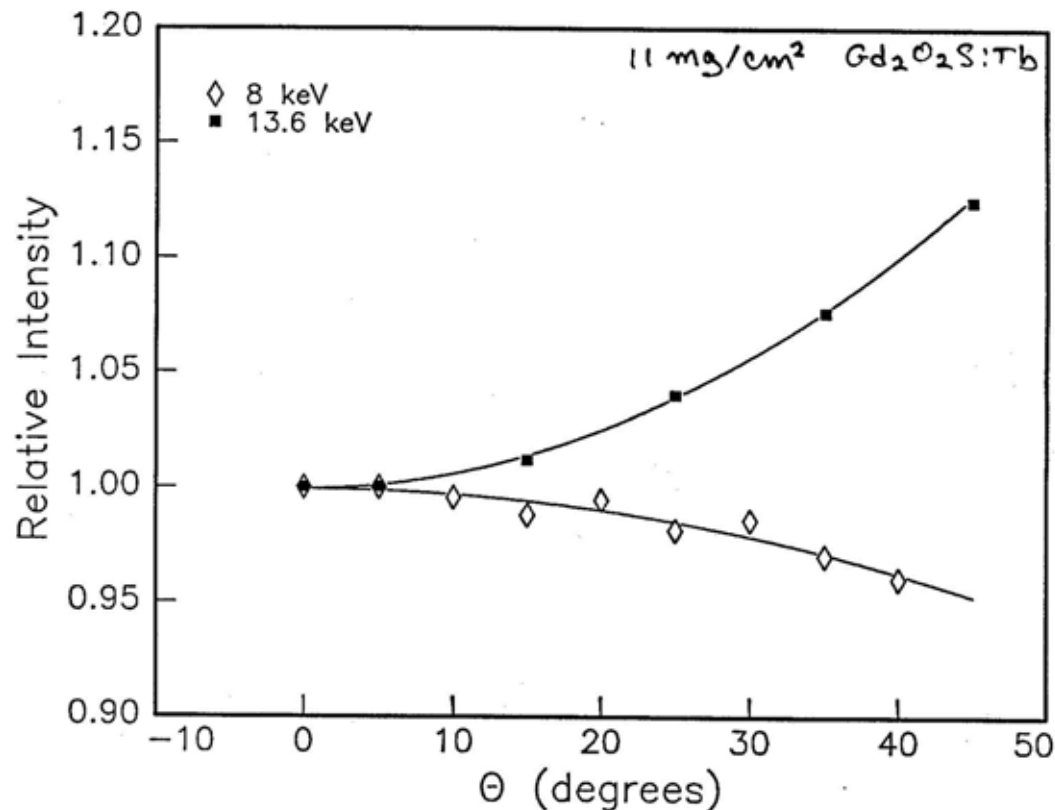
Settled Phosphors

The phosphor sensitivity depends on both the angle of incidence of the x-ray to the screen surface and the energy of the x-ray. These two effects have opposite signs:



Settled Phosphors

The resultant sensitivity of the phosphor screen varies slowly and smoothly with x-ray energy and angle of incidence. Because the behavior is slowly varying, a small number of data points map out the 2-dimensional functional surface of sensitivity vs. angle and x-ray energy and may be used to calibrate the phosphor response. This surface is readily fit to a surface quadratic in energy and angle. The coefficients of this fitted surface may be readily used to compute the response. (From Gruner et al, *Proc SPIE*, 2009 (1993) 98)



Fiber Optics

Fiber Optic Bundles

An glass optical fiber consists of a glass core of index N_2 surrounded by a glass cladding of index N_3 : As long as the core index is higher than the clad index, there will be a range of incident angles which result in total internal reflection of the light \Rightarrow propagation of light down the fiber. Total internal reflection can be very efficient, with losses of less than 0.000001/reflection, as compared to aluminum mirror which might lose 0.1/reflection!

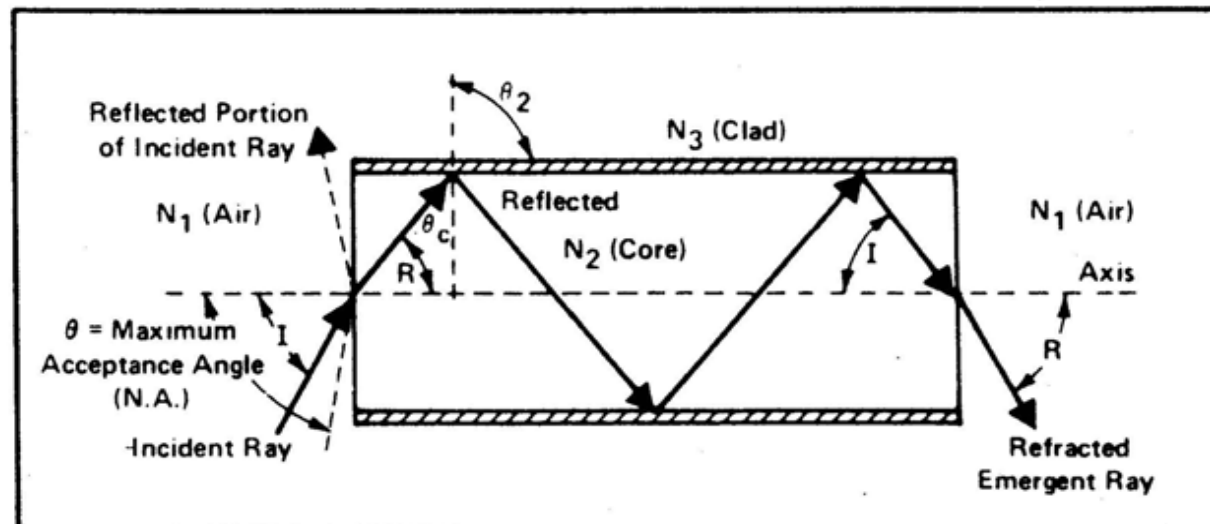


Figure 1 *Refraction, Reflection, and Numerical Aperture*

From W.P. Sigmund, "Fiber Optics." In *Handbook of Optics*, W.G. Driscoll & W. Vaughan, eds. (McGraw-Hill, NY, 1978).

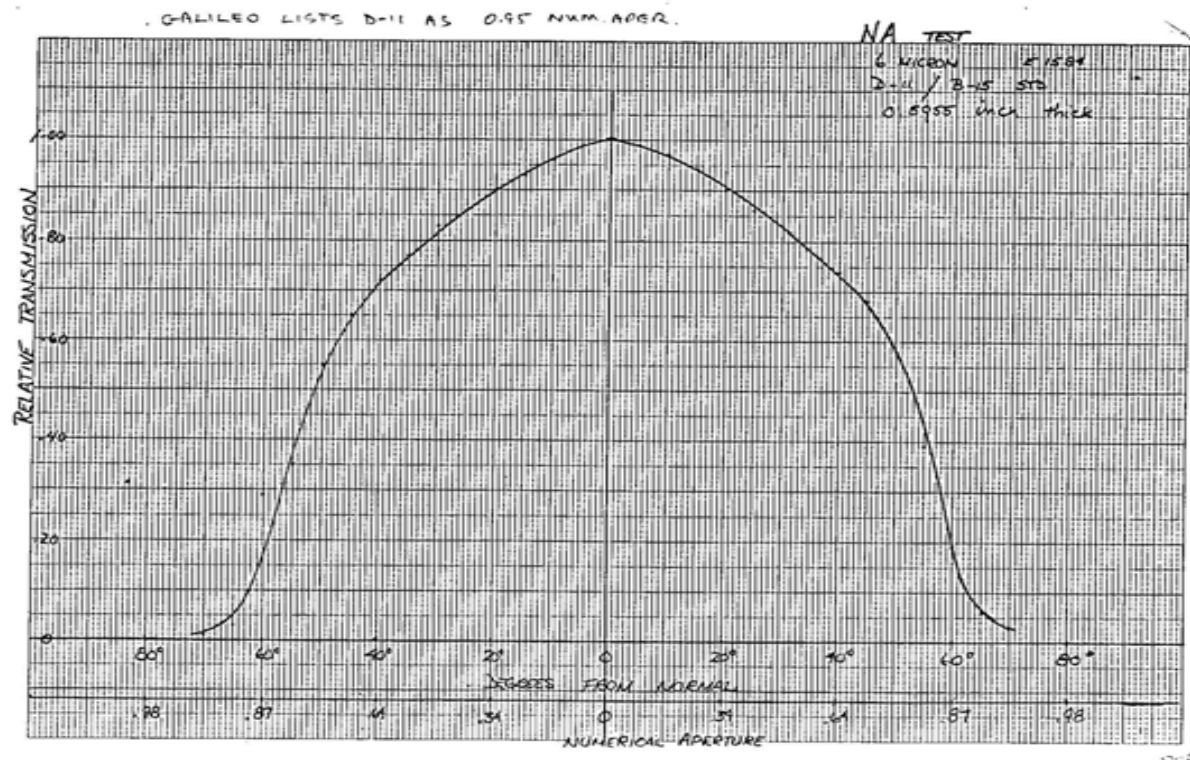


Fiber Optics

By Snell's law, the maximum angle for acceptance of light into the fiber is given by

$$N_1 \sin \theta_{MAX} = (N_2^2 - N_3^2)^{1/2},$$

where $\sin \theta_{MAX}$ is called the Numerical Aperture (NA). Now a days, NA=1 fiber optics are available, which is simply amazing: NA=1 fibers will accept light right up to 90° to the surface! The following graph shows the transmission of a fiber optic plate of NA=0.87 fibers.



Fiber Optics

Glass fibers may be drawn down to micron diameters, stacked side-by-side and fused into a coherent bundle of glass. If the ends of this bundle are polished, the fiber optic bundle will transmit light with amazing efficiency from one side of the bundle to the other. The resolution of the bundle is then set primarily by the fiber size. It is also set by light scatter from defects in the fiber, since a light ray which is scattered beyond the critical angle will single be transmitted across the fibers. To prevent this, modern fiber optic bundles incorporate black, light absorbing fibers between the light transmitting fibers. This is called Extra Mural Absorption or EMA. Fiber optic bundles without EMA are almost useless.

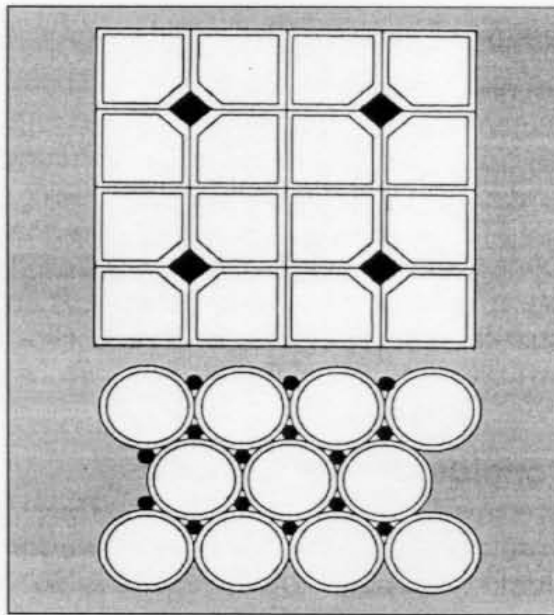
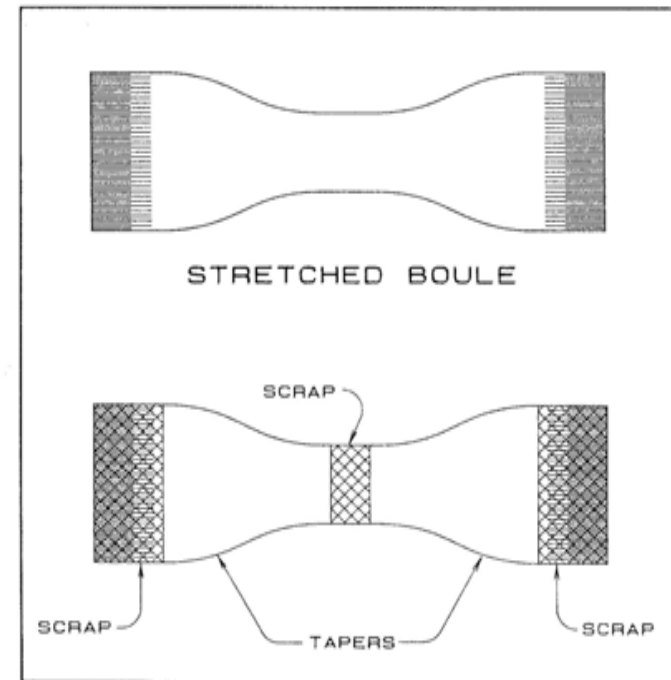
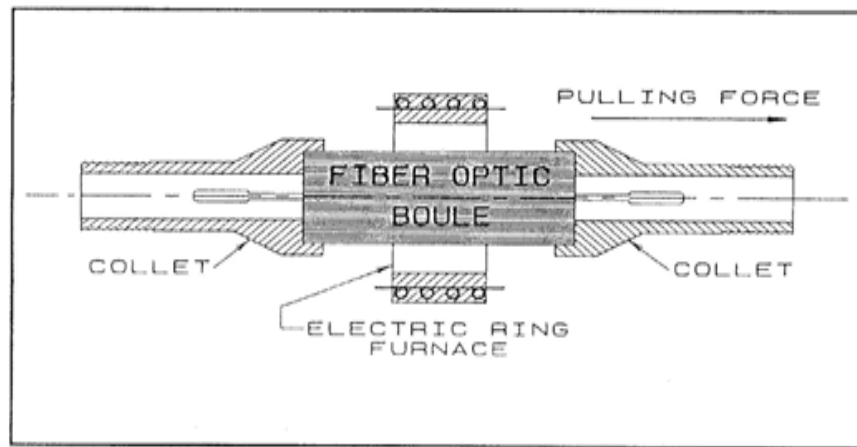


Figure 5: Square Pack and Hex Pack with Interstitial EMA



Fiber Optics

A fiber optic bundle, or boule as it is called, may also be drawn and cut to create a fiber optic taper.



Thanks to Schott Fiber Optics, Inc.



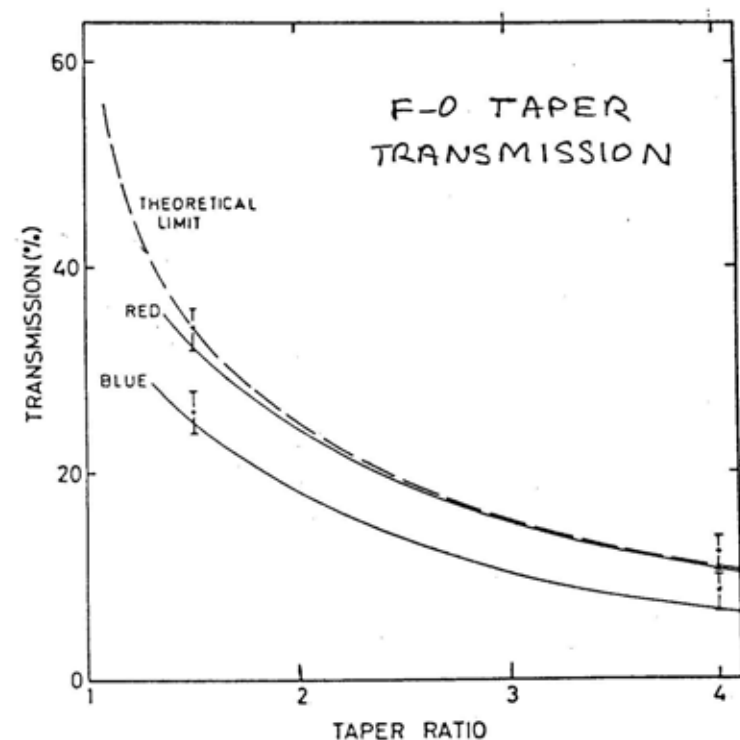
Fiber Optics

Why do this? Fiber optic tapers are amazingly efficient. Recall that a good phosphor might be, say, 15% efficient in converting x-ray energy to visible light. So for an 8 keV x-ray converting in a green emitting phosphor, one might get

$$\frac{8000 \text{ eV/x-ray}}{2.5 \text{ eV/green photon}} \times 0.15 \approx 500 \text{ photons,}$$

half of which go the wrong way. So one might get, say, 250 photons/x-ray. CCDs are usually small and desired x-ray detective areas are frequently large, so there is a need to demagnify the phosphor screen image. We could demagnify either with lenses or with fiber optic tapers.

Fiber optic tapers are close to the ideal set by the brightness theorem, e.g., the product of fiber area and divergence on the input is nearly the same at the output:



C.I. Coleman, *Adv. Electronics Electron Phys* **64B** (1985) 649.



Fiber Optics

Lenses are much worse, simply due to solid angle subtended for a given focal length. The lens coupling efficiency for magnif. $M = \frac{\text{Image}}{\text{Object}}$ is

$$\left[\frac{M}{2f(1+M)} \right]^2, \text{ where } f = \text{lens } (1/f).$$

| M | F.O. taper effic. | (f/1.0) lens effic. |
|------|-------------------|---------------------|
| 1 | 75% | 6% |
| 0.5 | 20% | 3% |
| 0.3 | 13% | 1.3% |
| 0.25 | 9% | 1% |

Coleman, 1985

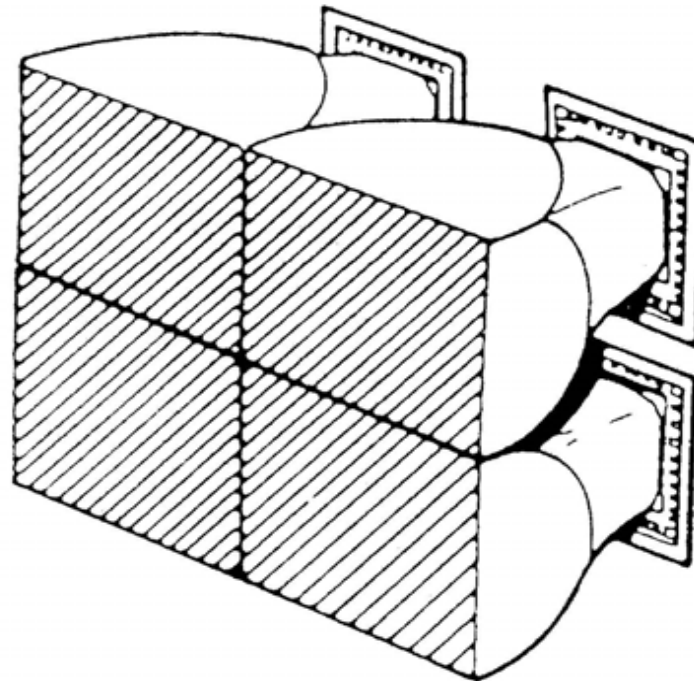
For 4:1 demagnification, coupling the phosphor to the CCD by a f/1.0 lens would result in about $1\% \times 250 = 2.5$ photons/x-ray onto the CCD. The CCD has a quantum efficiency of, say, 30%, so we get less than one e-h pair / x-ray, which stinks, given that the noise per pixel of a good, slowly scanned, cooled CCD is 5 electrons. But if we use a taper, we get $9\% \times 250 = 22.5$ photons/x-ray onto the CCD. Times 30% CCD QE and we get about 7 e-h pairs/x-ray, which gives a per pixel S/N of unity. So by using FO-tapers we can get excellent efficiency even with 4:1 demagnification



Fiber Optics

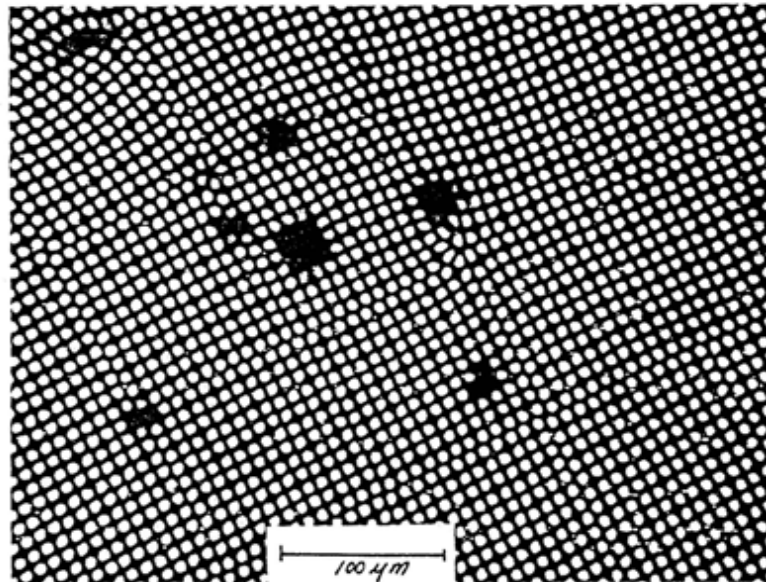
Fiber optic tapers suffer from some problems, though, which makes FO taper specification a job only for the experienced:

- FO manufacture is a black art and the world's supply comes from just a few small firms, most of which are within a few miles of Sturbridge, MA. The employees play musical chairs.
- FO tapers are available up to about 160 mm at the wide end, but these are heavy and expensive. The solution, which is expensive and complex, is to build a CCD mosaic:



Fiber Optics

- High resolution, high efficiency tapers require high NA fibers and excellent EMA. The best options are not available in large tapers. EMA performance is very dependent upon the type of glass.
- Rare earths are used in making the FO. These carry actinide contaminants which cause “zingers”, which are radioactive decay excitations of the phosphor and CCD.
- FO boules consist of a hierarchy of stacked fiber bundles. Defects in between the substructures are visible as “chickenwire” and shears
- Broken fibers and bubbles lead to measles.

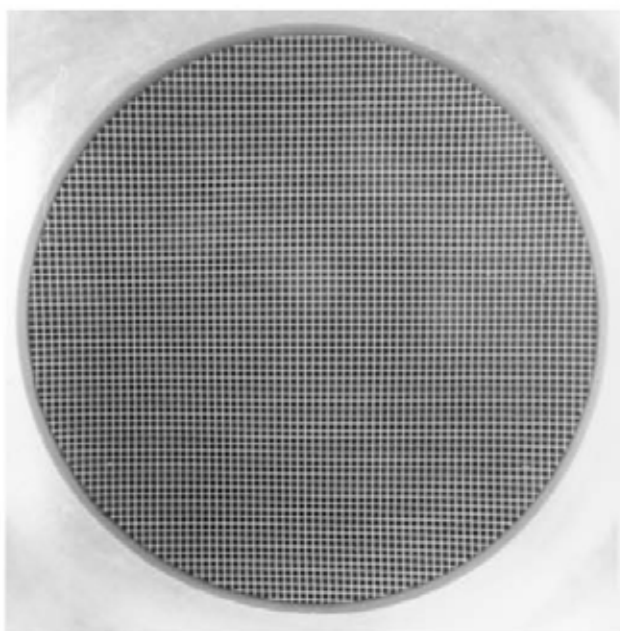


Deckman & Gruner, *Nucl Inst Meth* 246 (1986) 527..

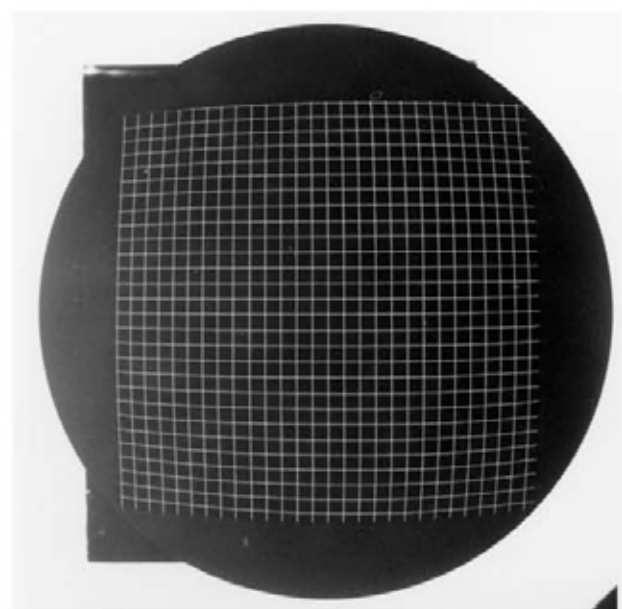


Fiber Optics

Tapers suffer from gross distortion



Small Taper

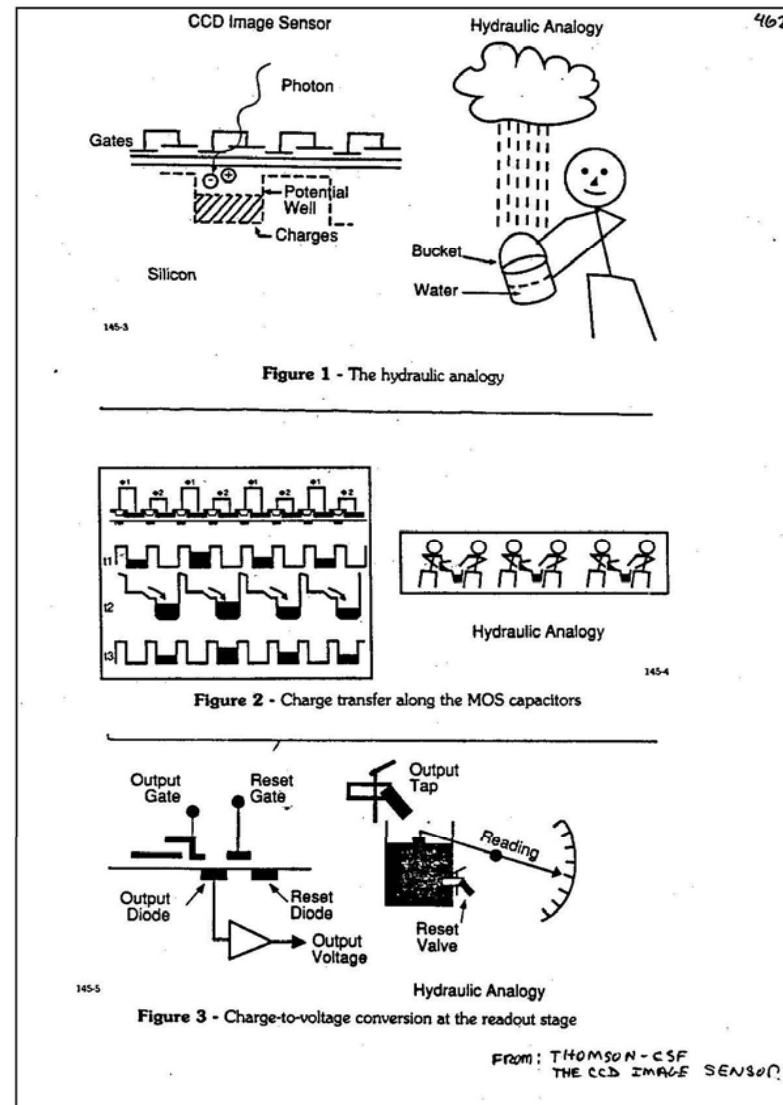


Large Taper



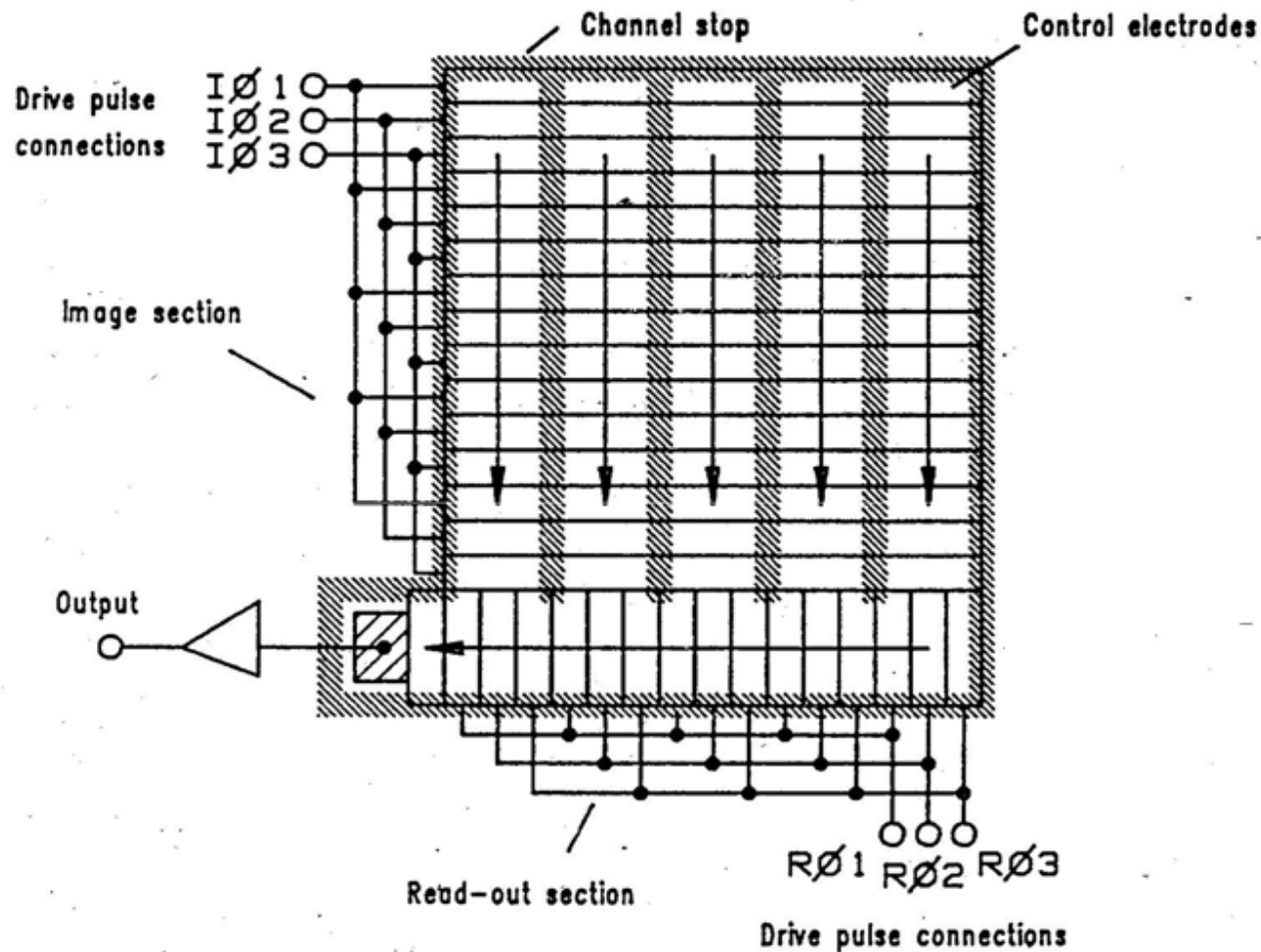
CCDs

The CCD is an astonishing, quantum limited device. Noise levels of 1-2 electrons have been demonstrated.



CCDs

CCDs work by charge shifting, which permits the neat trick of making a fast 1-dimensional detector.



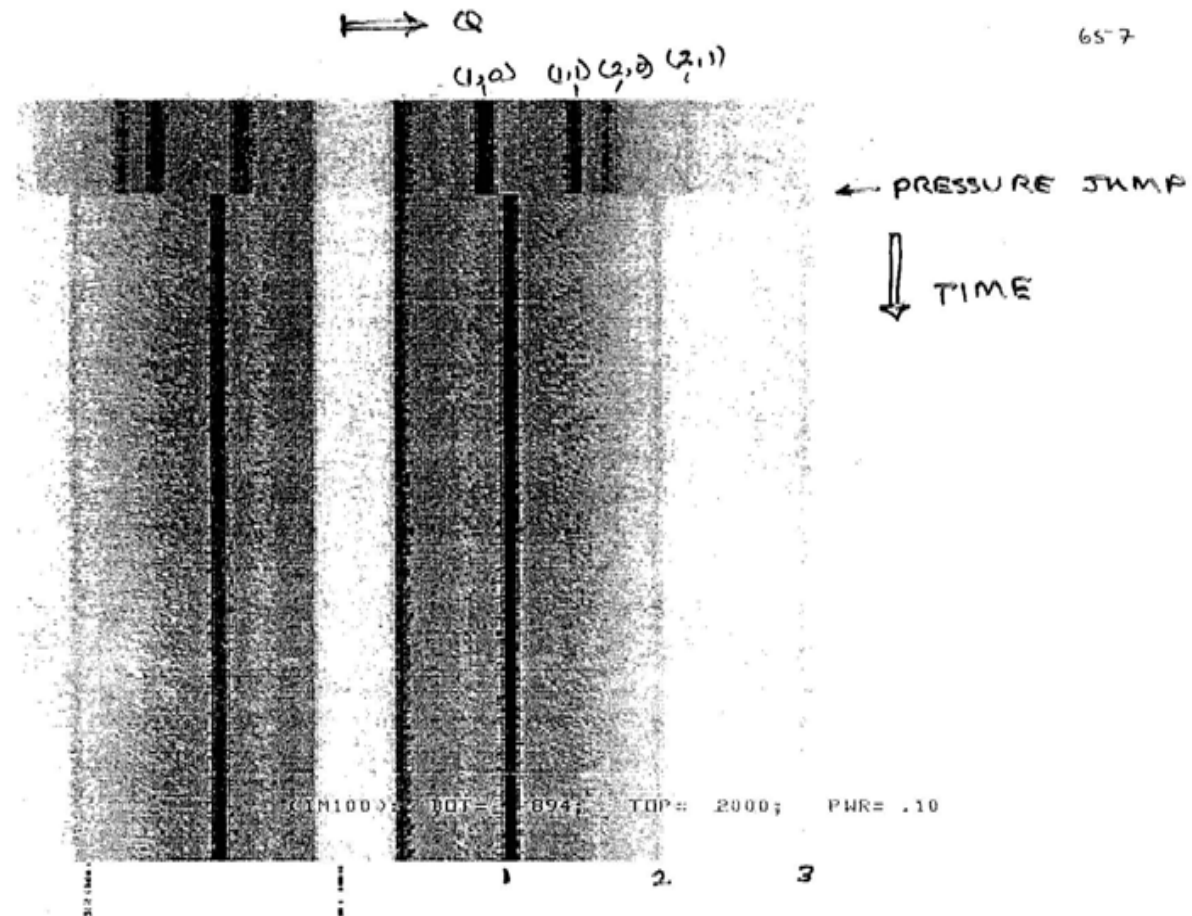
From: Burt, *Nucl Instr Methods* A305 (1991) 564.



CCDs

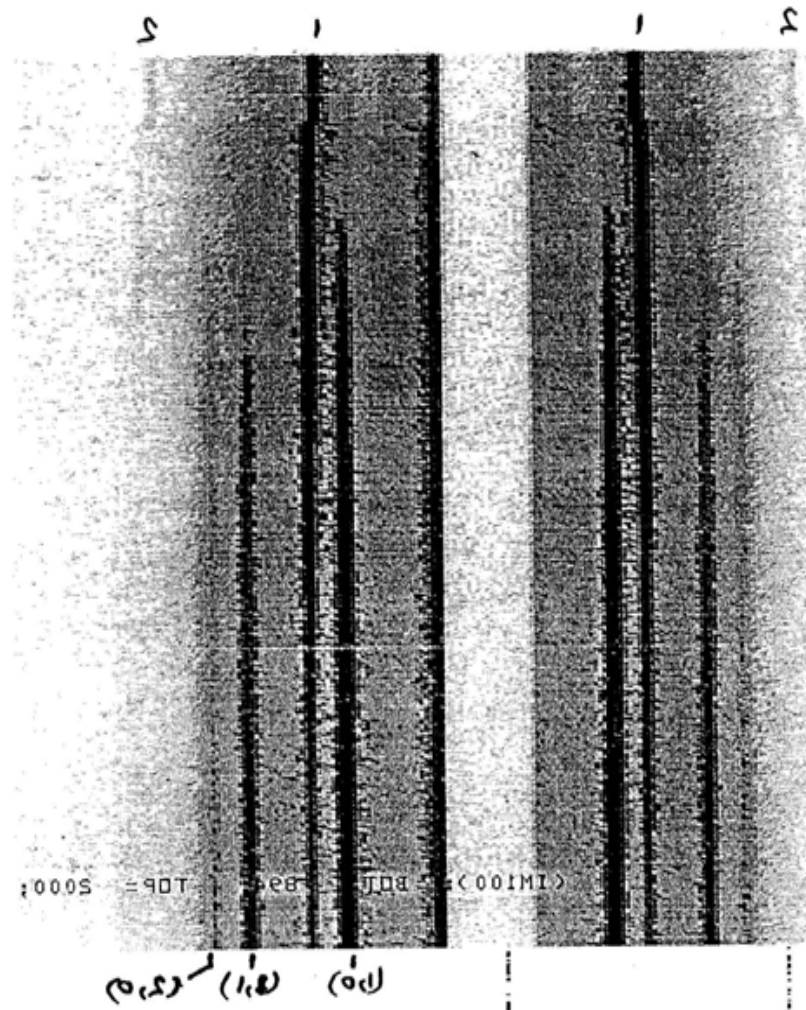
An example of the use of charge shifting is the following time-resolved pressure-jump experiments performed at the NSLS (see Osterberg et al, 1994; Erramilli et al, 1995):

DOPE, excess water, $H_{II} \rightarrow L_{\alpha}$, 25 C, 102 bar \rightarrow 1935 bar, 9 msec/line. NSLS X-9.



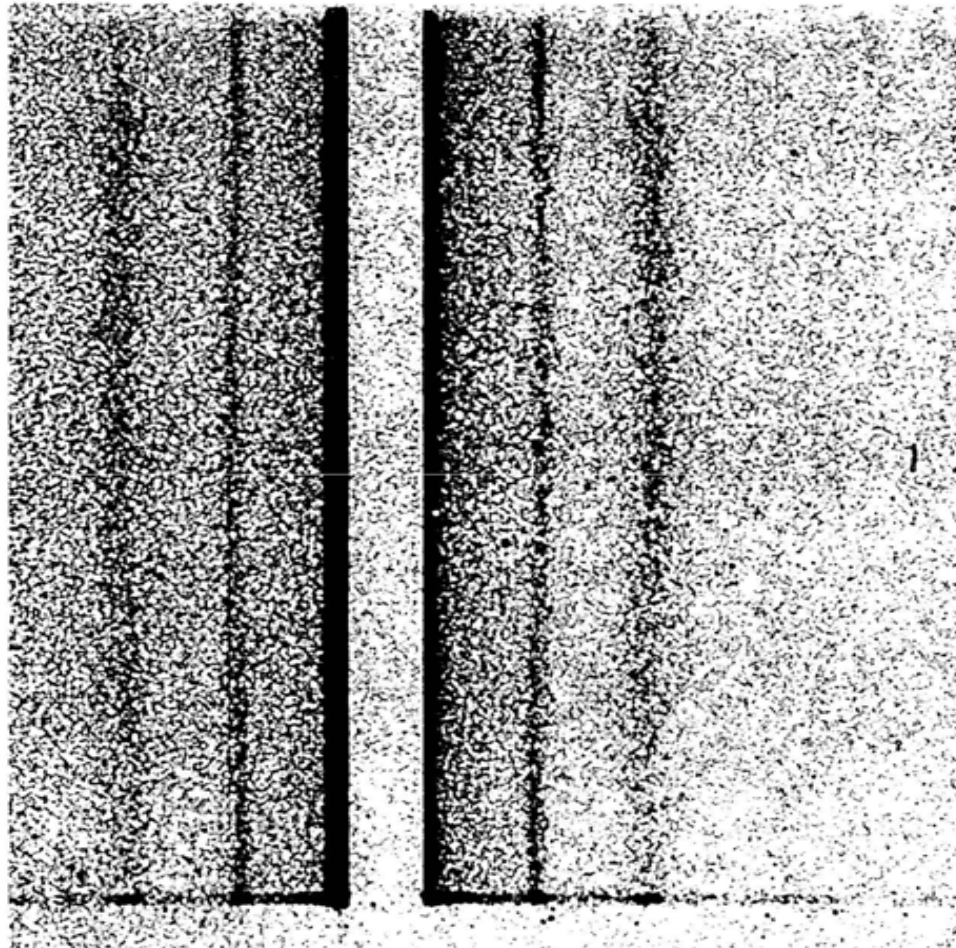
CCDs

DOPE, excess water, $H_{II} \rightarrow L_{\alpha}$, 25 C, 1895 bar \rightarrow 222 bar, 9 msec/line



CCDs

Frog sartorius muscle twitch. CHESS, 1 msec/line. The change in the layer line spacing is the observation of interest. (E. Eikenberry)



CCD Considerations

- Format (e.g., 1k x 1k)
- Pixel size, typically $(20 - 25 \mu\text{m})^2$
- MPP vs. non-MPP
- Blooming
- Read-out Speed
- Single vs. Multi-port CCDs
- Operating Temperature
- Controller flexibility

Considerations are constrained by CCD availability and FO-bonding options.



CCD Detector Design Handbook

- 1) Phosphor efficiency (x-ray energy/light energy) $\approx 10\%$.
Conservatively, given collection losses, say 5%.

Example:

$$\frac{10 \text{ keV} \cdot 5\%}{2.5 \text{ eV/photon}} \approx 200 \text{ photons.}$$

- 2) Lens coupling efficiency for magnification, $M = \frac{\text{Image}}{\text{Object}}$

is $\left[\frac{M}{2f(1+M)} \right]^2$, where $f = \text{lens } (1/f)$.

- 3) Fiber optic tapers do better:

| M | F.O. taper effc. | $(f/1.0)$ lens effc. |
|------|------------------|----------------------|
| 1 | 75% | 6% |
| 0.5 | 20% | 3% |
| 0.3 | 13% | 1.3% |
| 0.25 | 9% | 1% |



CCD Detector Design Handbook

- 4) Scientific CCDs in slow-scan, cooled mode conservatively have
 - ~ 10 e- noise/pixel
 - $\sim 30 - 40\%$ quantum efficiency

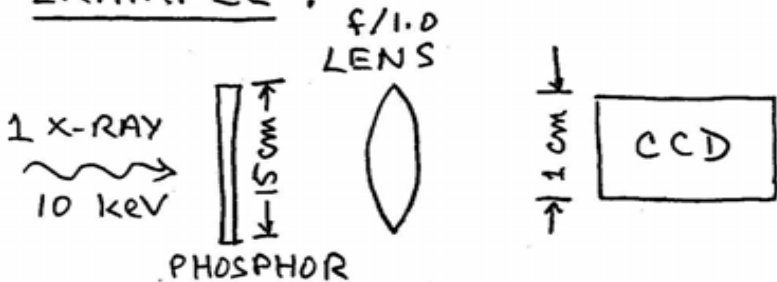
5) **Details Count!!**



CCD Detector Design Handbook

Let's try some real design examples:

EXAMPLE :



1 10 keV X-RAY YIELDS 200 PHOTONS
 LENS EFFICIENCY = 0.001
 \therefore PHOTONS INCIDENT ON CCD
 $= 200 \times 0.001 = 0.2 \text{ /X-RAY}$
 FOR 40% QUANTUM EFFIC. IN
 CCD, EACH X-RAY YIELDS

$$0.2 \frac{\text{PHOTONS}}{\text{X-RAY}} \times 0.4 \frac{e^-}{\text{PHOTON}} = 0.08 \frac{e^-}{\text{X-RAY}}$$

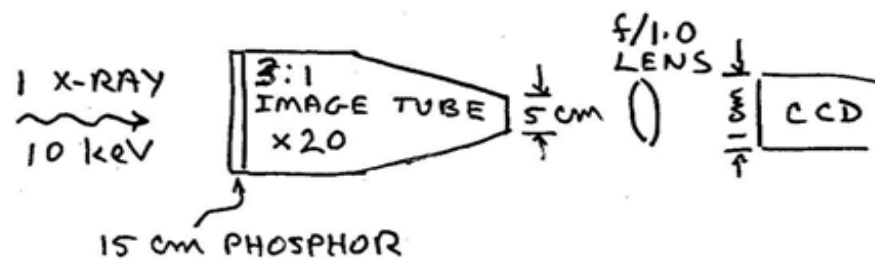
$$\frac{\text{SIGNAL}}{\text{NOISE}} \Big|_{\text{PIXEL}} = \frac{0.08}{10} = 0.008$$

BAH!



CCD Detector Design Handbook

EXAMPLE :



$$\begin{aligned}
 &1 \text{ 10 keV X-RAY} \Rightarrow 200 \text{ PHOTONS} \\
 &\quad \times (\text{IMAGE TUBE GAIN OF 20}) = 4,000 \text{ PHOTONS} \\
 &\quad \times (\text{LENS EFFIC.} \approx 0.007) = 28 \text{ PHOTONS} \\
 &\quad \times (40\% \text{ CCD QUANTUM EFFIC.}) = 11 \frac{e^-}{\text{X-RAY}}
 \end{aligned}$$

$$\frac{S}{N} = \frac{11}{10} \approx 1.$$

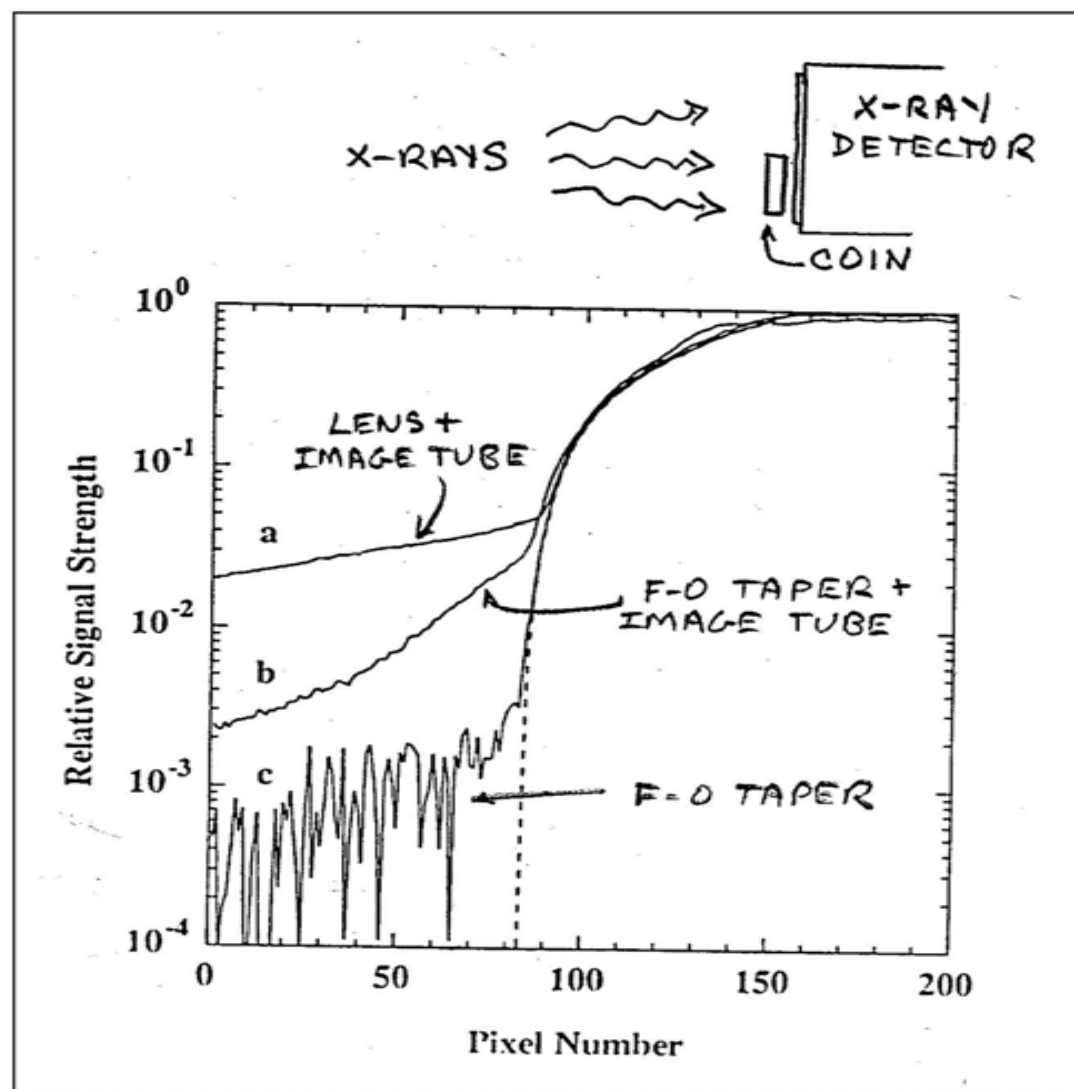
BETTER, BUT

- a) NEED IMAGE TUBE
- b) GET SIGNAL INDUCED NOISE.



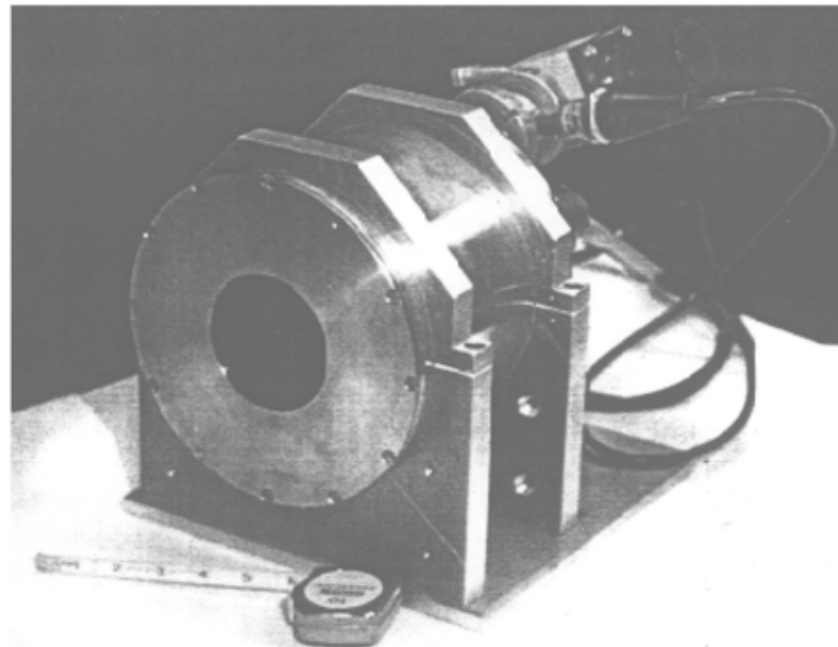
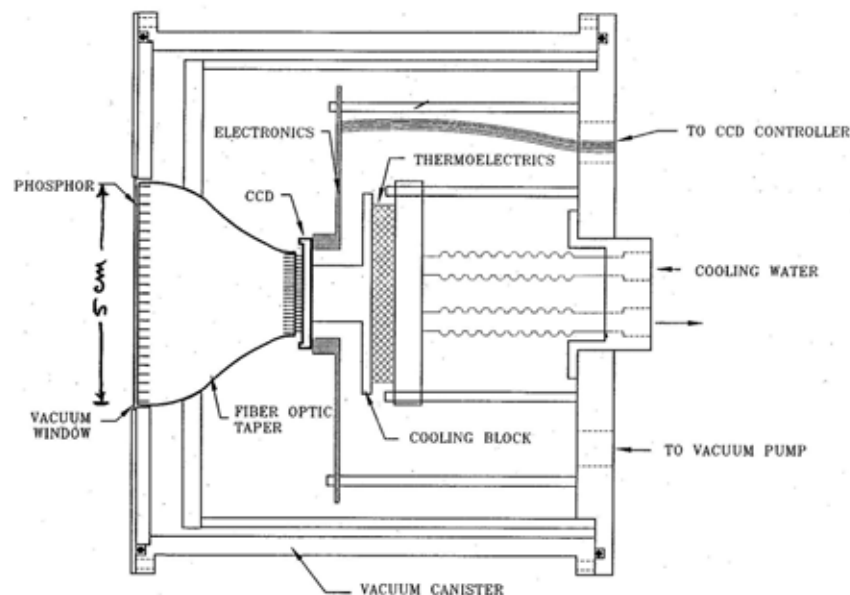
CCD Detector Design Handbook

The "coin" test is a simple test for signal induced background.



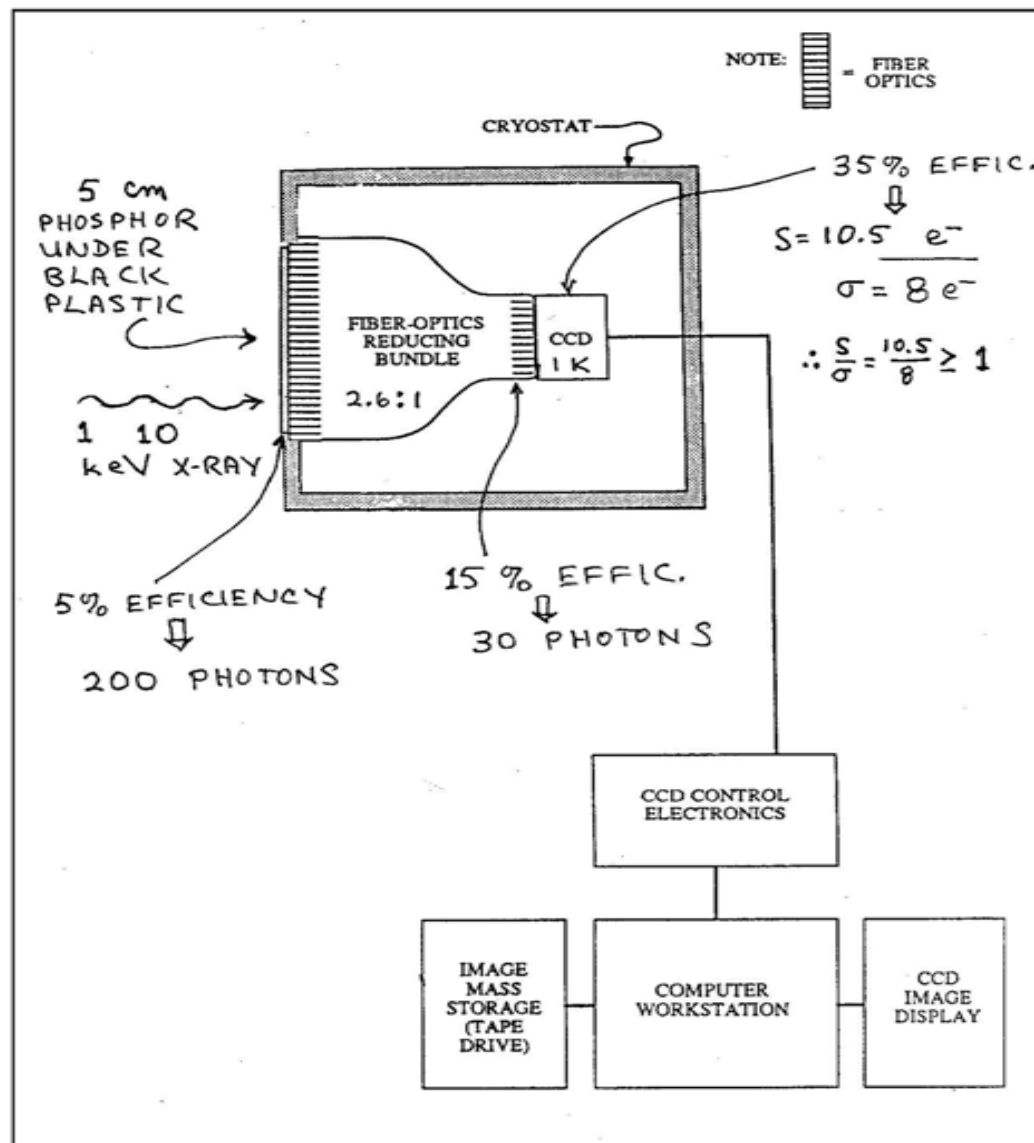
CCD Detector Design Handbook

The “1k detector” served as a very successful prototype for the generic CCD designs which followed (Tate et al, *J Appl Cryst*, 28 (1995) 196. It has been the main data acquisition instrument of an enormous number of experiments.



CCD Detector Design Handbook

It's quantum chain:



CCD Detector Design Handbook

| | |
|---|-------------------------------------|
| CCD | Thomson TH7896AVRNF |
| Pixel format | 1024 × 1024 |
| Fiber optic reduction ratio | 2.6:1 |
| Active input area | 51 × 51 mm ² |
| Pixel size at phosphor (microns) | 50.1 |
| Phosphor | Gd ₂ O ₂ S:Tb |
| Operating temperature | -60 °C |
| A/D resolution (bits) | 16 |
| Gain (e ⁻ /ADU) | 4.6 |
| Sensitivity (e ⁻ /5.9 keV x-ray) | 4.6 |
| Read noise (e ⁻ /pixel) RMS | 8 |
| Dark accumulation (e ⁻ /pixel/s) | 0.8 at -60 °C |
| Full well (e ⁻ /pix) | 4 × 10 ⁵ |
| Point spread (microns) | |
| full width half maximum | 80 |
| full width tenth maximum | 165 |
| full width hundredth maximum | 230 |
| FW 0.1% M | 450 |

TABLE II
Detector sensitivity vs. x-ray energy

| X-ray energy (keV) | 5.9 | 8.0 | 8.9 | 11.0 | 13.5 | 18.0 |
|---|-------|-------------------------|-------------------------|------------------------|------------------------|------------------------|
| Fraction of x-rays stopped ¹ | 0.932 | 0.986 | 0.975 | 0.878 | 0.703 | 0.430 |
| Signal/incident x-ray (e ⁻) | 4.6 | 7.1 | 7.7 | 10.3 | 11.1 | 10.3 |
| Signal/stopped x-ray (e ⁻) | 4.9 | 7.2 | 7.9 | 11.7 | 15.8 | 24.0 |
| Signal/stopped x-ray/Energy (e ⁻) | 0.83 | 0.90 | 0.89 | 1.06 | 1.16 | 1.33 |
| Quadratic response coefficient ² | — | -3.5 × 10 ⁻⁵ | -2.2 × 10 ⁻⁵ | 2.0 × 10 ⁻⁵ | 6.0 × 10 ⁻⁵ | 1.2 × 10 ⁻⁴ |

¹ For 11.5 mg/cm² Gd₂O₂S:Tb phosphor.

² Quadratic coefficient, B, characterizing the change in detector response, R, as a function of angle as
R = 1 + B × θ², where θ is given in degrees.

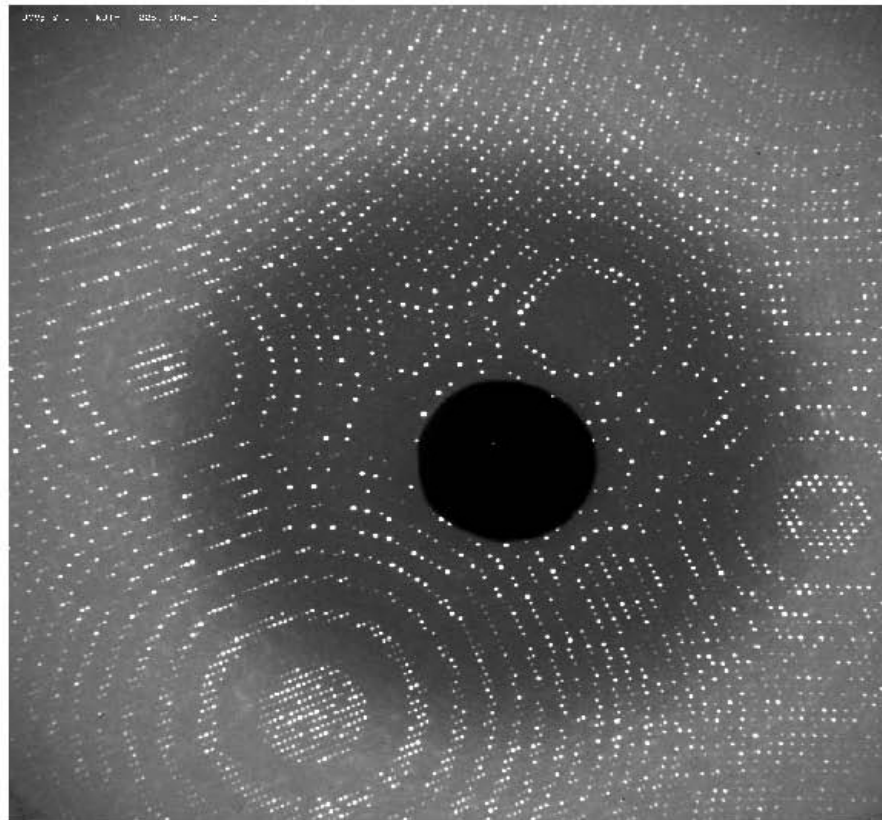
TATE et al, J. APPL. CRYST. 28 (1995) 196.



“1-k Detector”

This is an example of a diffraction pattern taken at the CHESS F-1 station using the 1k detector. The actual size of the diffraction pattern on the detector face is 5 cm across.

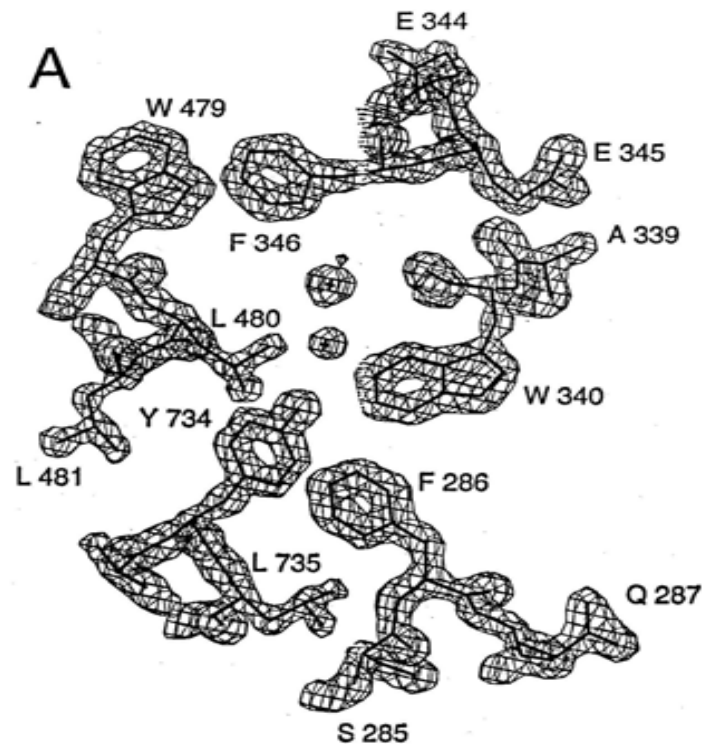
Tomato bushy stunt virus. F-1, 100 micron collimator, I_23 space group, 365 Å unit cell, 0.2° oscillation, 40 sec exposure. (Courtesy of D. Rodgers & S. Harrison)



“1-k Detector”

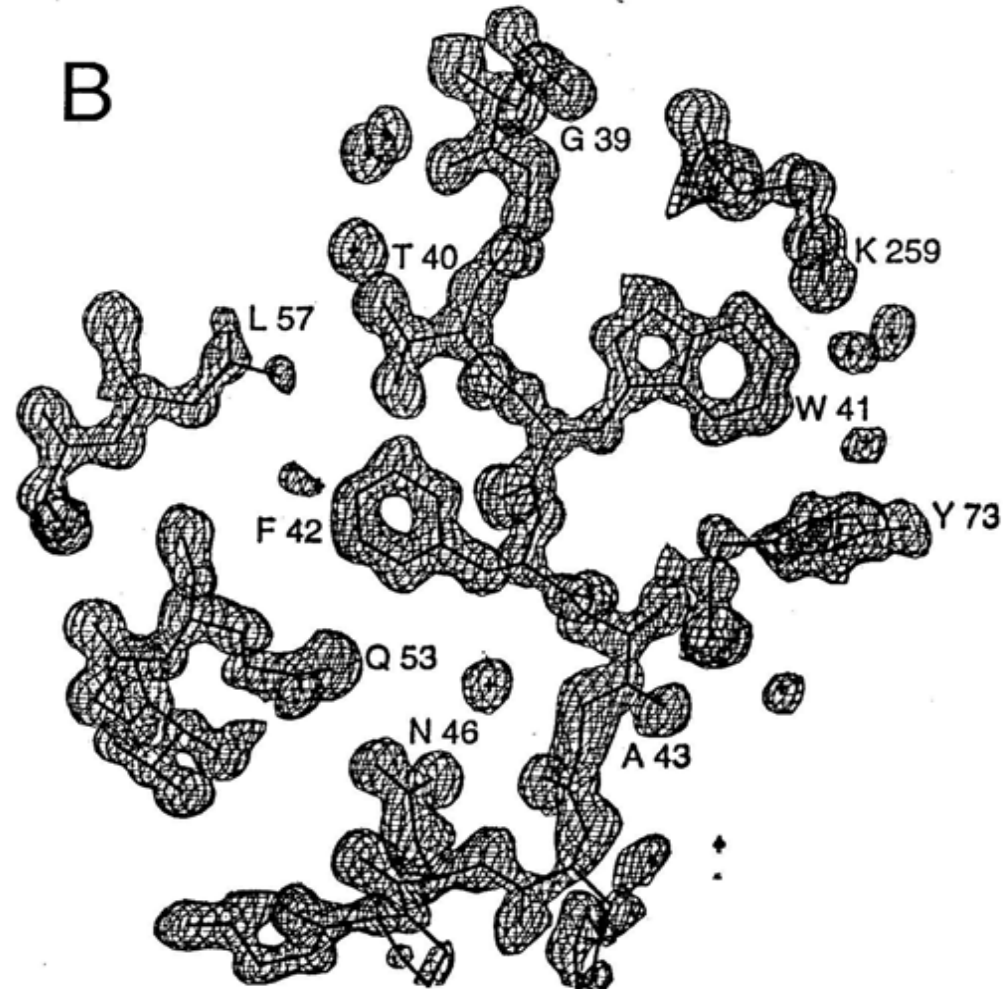
The accuracy of the 1k-detector led to electron density maps which were quite extraordinary for their time and finally convinced the community that CCD detectors could deliver data comparable to photon counters (Figures from Walter et al, 1995)

$2|F_0 - F_c|$ electron density map of lipoxxygenase at 1.4 Å resolution. 839 amino acids, P2. 96% complete; 1,135,803 observations, $R_{\text{SYM}} = 3.6\%$ to 1.4 Å The data set was taken in 1 evening at CHESS by Wladek Minor.



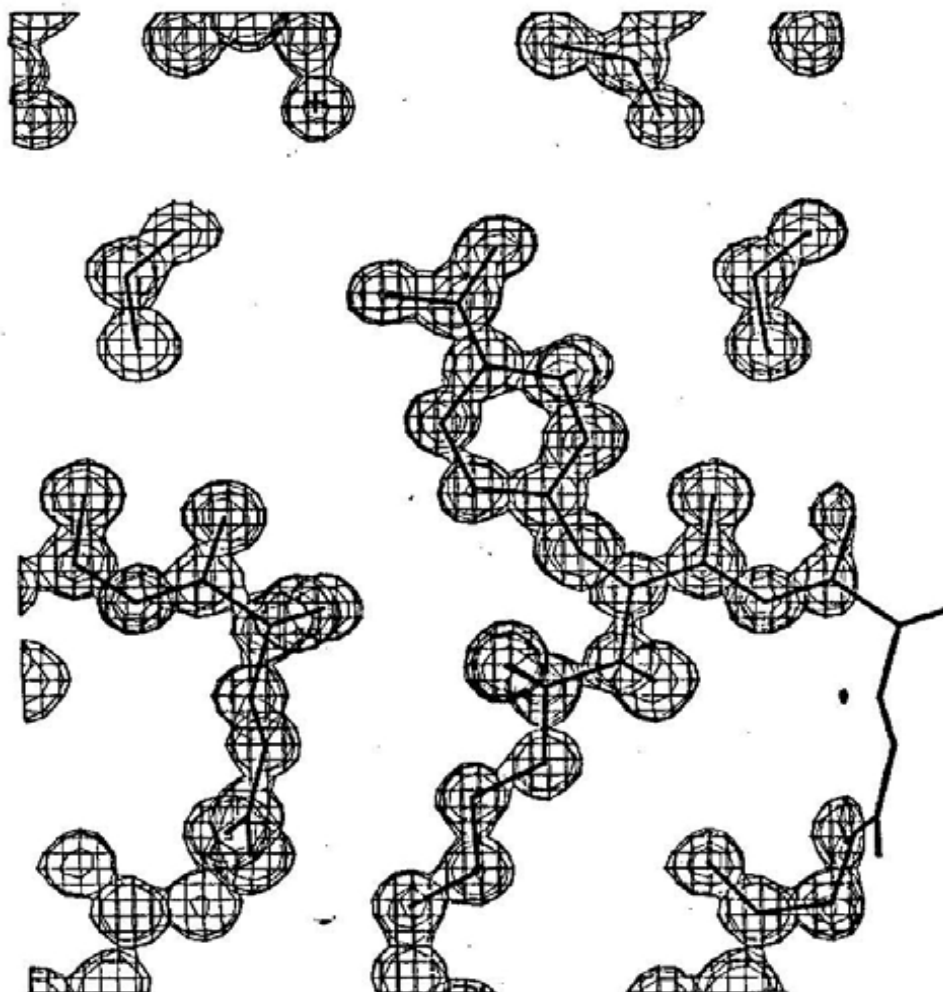
“1-k Detector”

Catalytic domain of Cellulase E2 at 1 Å resolution. 70% complete, $R_{\text{SYM}} = 9.1\%$.
CHESS. Data by J. Sakon & P.A. Karplus.



“1-k Detector”

Ossamycin, CHESS F-2, 25 keV x-rays, 1.0 Å resolution, direct refine method. Data by E. Lobkovsky & J.C. Clardy.



CCD Dark Current

The dark current on a well-cooled CCD detectors is sufficiently low that long exposures are possible, even if several frames need to be summed. The following example is of a tri-block co-polymer monodomain sample taken during an 8.9 hour exposure on a rotating anode generator.

Kraton D-1102 (pStyrene-pButadiene-pStyrene = 11k:58k:11k mol. wt.). Sample roll-cast from cumene by the Ned Thomas group. Data by D. Hajduk & S. Gruner.

