

## HIGH RESOLUTION TOMOGRAPHY WITH CHEMICAL SPECIFICITY

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A very fast method of computerized critical absorption tomography featuring  $\sim 10 \mu\text{m}$  spatial resolution and high chemical sensitivity is described. Synchrotron radiation is used and the method is especially suited to investigating small samples. From a preliminary experiment it is found that layers of neighboring elements only  $0.2 \mu\text{m}$  thick can be distinguished at medium atomic numbers.

## 1. Introduction

In recent years, computerized tomography (CT) [1] has gained increased interest, mainly because significant improvements have been made in detectors, sources and computers. At the same time, CT has been more frequently applied in nonmedical fields such as general materials research [2,3] or even forestry [4]. For the particular case of CT of small samples, Grodzins [5,6] has investigated how, with a minimum of measuring time, a maximum of spatial resolution and/or chemical sensitivity is obtained. In agreement with some general expectation it follows from Grodzins' study that synchrotron X-radiation (SR) would be best suited to CT of small samples.

Because SR is very bright, very intense and easily tunable, CT employing SR can be shown to be superior to CT with conventional X-rays, especially for small samples. In this study we demonstrate the virtues of synchrotron radiation for performing tomography with critical absorption identification of single elements and spatial resolution of  $\sim 1 \mu\text{m}$ . With appropriate perfect crystal optics combined with a two-dimensional position sensitive detector, fast, high resolution CT of small samples with good spatial and chemical resolution becomes feasible at short exposures. For example, at a source like DORIS operated at 3 GeV, 100 mA, bending magnet radiation, a model specimen of 3 mm diameter and 25 mm length would require  $\sim 6$  min to image at a  $25 \mu\text{m}$  cube voxel size.

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## 2. Parallel beam method of computerized tomography

Using notations of fig. 1, an ordinary CT sample is scanned along  $x$  and  $z$  and rotated about the  $x$ -axis. Let the number of steps per scan in each direction be  $m = A/a = C/c$  and the angular increments of the rotation be  $180/m$  deg. The voxel size is then of the order  $a^3$  (if  $a = c$  may be assumed) and there are roughly  $m^3$  voxels within the sample [5]. At any fixed  $x$ -position ("x-slice"),  $m^2$  intensities are measured, each determined by the  $m$  voxels lined up behind each other for a fixed combination of angular and  $z$ -position of that slice. Any set of  $m$  voxels is subset of the  $m^2$  voxels present in the particular  $x$ -slice. Computer reconstruction of a slice thus requires solution of a set of  $m^2$  linear equations with  $m^2$  variables. For the whole sample consisting of  $m$  slices,  $m$  solutions have to be calculated, all independent of each other.

Fig. 2 illustrates how sequential  $x$ - and  $z$ -scan intensity measurements may be replaced by one simultaneous measurement. A wide beam of sufficiently small divergence combined with a two-dimensional array of charge coupled diodes (CCD) is used. Each CCD defines a pencil beam the intensity of which it measures. If the total measuring time in conventional CT is  $T$ , this time will be reduced drastically to  $T/m^2$ .

A beam with the required properties is obtained by combining an asymmetric with a symmetric Bragg reflection as shown in fig. 2. The asymmetry of the first reflection matches beam height to the height of the sample and at the same time reduced beam divergence, according to the laws of dynamical perfect crystal diffraction [7]. The symmetric reflection downstream does not increase beam divergence although the intrinsic acceptance angle of the second crystal is larger than the divergence of the beam incident onto it. The second

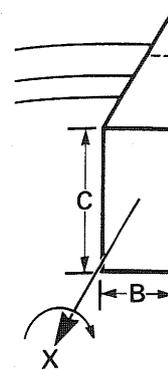


Fig. 1. Geometry

crystal simply produces a beam of the order of the total distance of 1 is  $1 \mu\text{m}$ .

Presently two size  $10 \times 10 \mu\text{m}$  we give the following needed to obtain above model specimen

Behind a detector in fig. 2, sources especially optimized of the order  $10^{-3}$  relative to photon fluxes of 1-5% detection counts/(s pixel) so measurement required. Assume measuring time energies. Allow the 300 angular

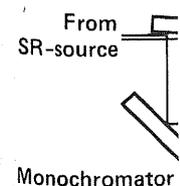


Fig. 2. Geometry for

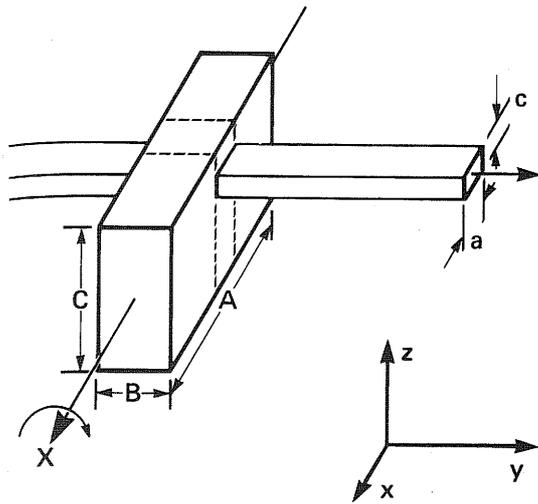


Fig. 1. Geometry for ordinary computerized tomography.

crystal simply passes the divergence it receives, typically of the order of  $10^{-4}$  rad. Thus, with a sample-to-detector distance of 10 mm, the blur due to beam divergence is 1  $\mu\text{m}$ .

Presently two-dimensional CCD detectors with pixel size  $10 \times 10 \mu\text{m}$  appear feasible [8]. For such a detector, we give the following estimate of total measuring time needed to obtain a two-wavelength CT image of the above model specimen of cylindrical shape.

Behind a double monochromator of the type shown in fig. 2, sources like DORIS or SPEAR, which are not especially optimized for this application, yield an intensity of the order of  $10^{10}$  photons/(s  $\text{mm}^2$ ) at roughly  $10^{-3}$  relative bandwidth. Thus, for a  $10 \times 10 \mu\text{m}$  pixel, photon fluxes of  $10^6 \text{ s}^{-1}$  are not unrealistic. Assuming 1-5% detection efficiency, we have  $10^4$  to  $5 \times 10^4$  counts/(s pixel). In this case  $m = 3 \text{ mm}/10 \mu\text{m} = 300$  so measurements at 300 different angular settings are required. Assuming 1% statistical error we need 300 s measuring time for one energy, or 10 min for two energies. Allowing an additional 300 s for performing the 300 angular settings per energy, the sample may be

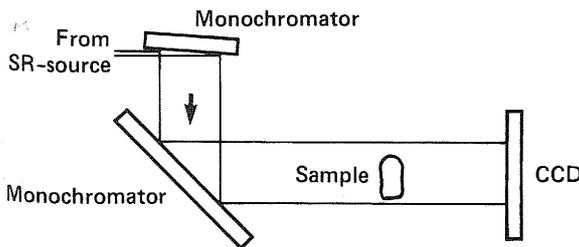


Fig. 2. Geometry for fast, high resolution tomography with SR.

imaged at 10  $\mu\text{m}$  resolution within 20 min. It should be noted that the huge number ( $2.25 \times 10^8$ ) of individual intensity measurements are performed in 20 min, that the sample is imaged at as many individual picture points (voxels), and that this result is obtained with no special sophistication with respect to the SR source. Furthermore, this estimate is conservative as regards detection efficiency and, moreover, does not outline the ultimate limits of the method.

### 3. Reduction of fluorescent and scattered radiation

An option of the above method yielding reduction of radiation that otherwise might cause blur in the imaging process is illustrated in fig. 3. The essential feature is a third diffracting, perfect crystal interposed between sample and detector. Deviated and/or inelastically scattered radiation on traversing the sample will no longer be reflected by the third crystal and thus be eliminated. This will apply to Rayleigh scattering at inhomogeneities within the sample, Compton scattered radiation, and fluorescent radiation. To what extent the deviated (blurring) radiation arises depends on the homogeneity and atomic number of the sample material and the energy of the imaging radiation in a more or less complex way and will vary considerably. With the third crystal, the sample-to-detector distance and thus the normal blur will necessarily increase, a compromise must be made between blur this and that caused by scattered radiation.

As indicated in fig. 3, in practice in order to simplify the alignment, second and third crystals would be manufactured as parts of a single monolithic crystal. An alternative for reduction of unwanted radiation is to place the sample between the first and second crystals and position the detector close to the second crystal.

It is clear that rejection of unwanted radiation is not complete. First, a ray that is scattered normal to the

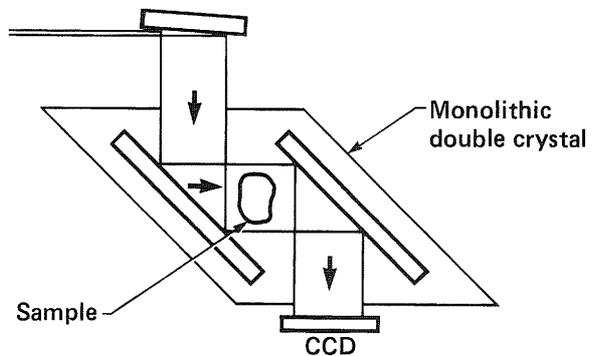


Fig. 3. Geometry for high resolution tomography which minimizes undesired radiation.

plane containing incident and diffracted beams by an angle of the order of one minute or less is not prevented from being diffracted. Second, inelastically scattered radiation may still be diffracted at its appropriate Bragg angle which is different from the Bragg angle of the main beam. However, since Compton and fluorescent radiation are emitted into a large solid angle, only a small fraction will be Bragg reflected at the third crystal.

**4. Intensity and resolution estimate for six typical SR-sources**

It is interesting to study how different SR sources would perform with the parallel beam method of CT. Fig. 4 shows calculated fluxes (photons/s mm<sup>2</sup> at 20 m distance from the tangent point) behind a Ge 111 double monochromator for six typical SR-sources with electron energies ranging from 3 to 5.5 GeV and cur-

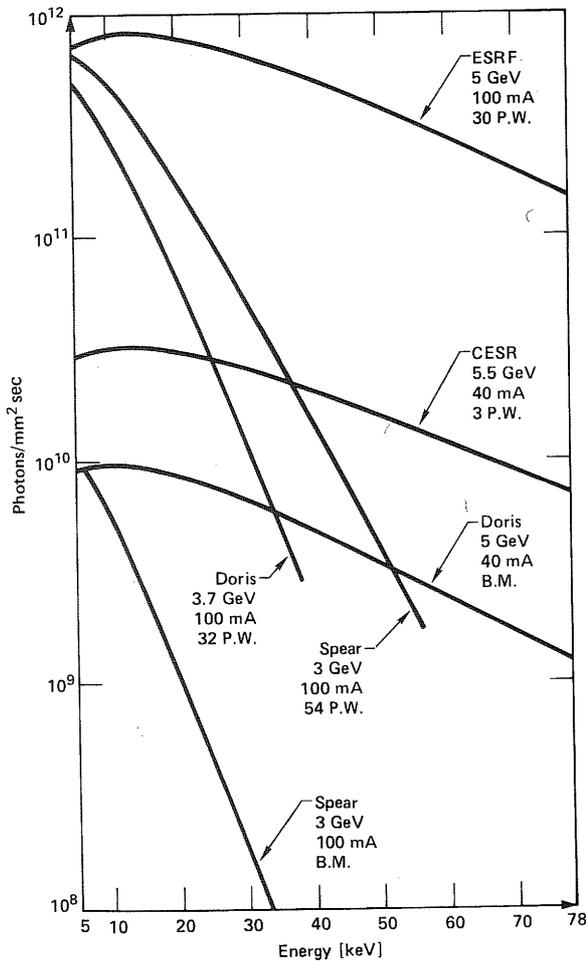


Fig. 4. Calculated fluxes for six synchrotron radiation sources.

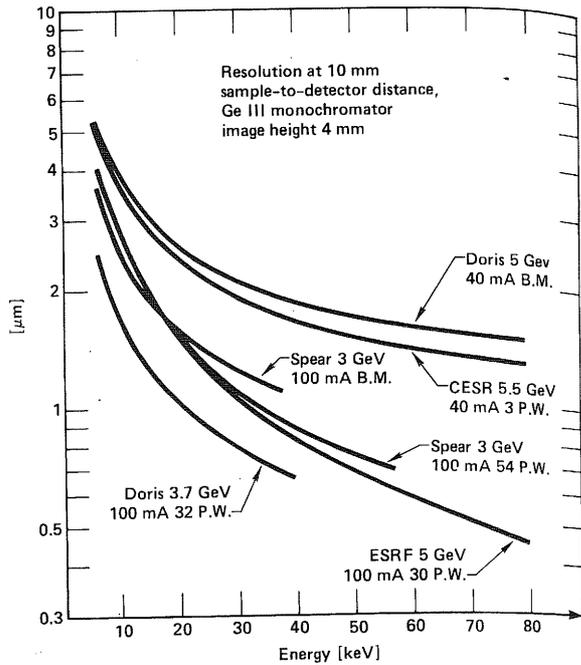


Fig. 5. Spatial resolution for six synchrotron radiation sources.

rents from 40 to 100 mA. Some are bending magnet (BM) and other *n*-pole wiggler (*n*PW) sources with *n* ranging from 3 to 54. The photon energies vary between 6 and 78 keV. In all cases the height of the imaging beam is normalized to 4 mm. As seen for energies up to 10 keV, fluxes with all sources do not differ by more than a factor of 100. At 35 keV this factor has increased

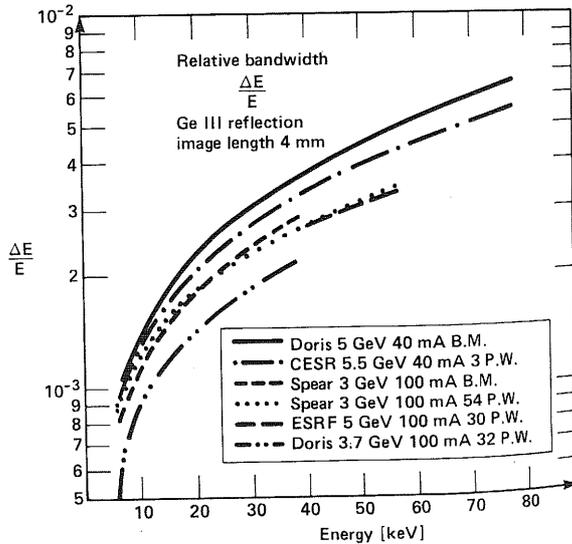


Fig. 6. Relative bandwidth for six synchrotron radiation sources.

to almost 10<sup>4</sup>. At energies of 5 GeV claimed above. Wiggler sources over Fig. 5 gives 10 mm sample-determined by natural beam diameter between 5 and 0.5

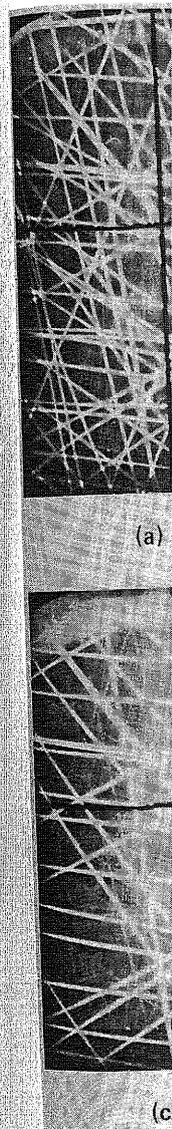


Fig. 7. (a) Web of diffraction spots above Cu K-α, (c) above Ni K-α.

to almost  $10^4$ . Near 78 keV only sources with electron energies of 5 GeV and more provide the high speed claimed above. The great advantage of multipole wiggler sources over BM sources is also evident from fig. 4.

Fig. 5 gives the corresponding spatial resolution at 10 mm sample-to-detector distance in so far as it is determined by the properties of the source size and natural beam divergence. As is seen, a resolution between 5 and  $0.5 \mu\text{m}$  is quite feasible. If necessary, it

could be reduced by placing the sample closer than 10 mm to the detector. In most cases this is unlikely to be necessary.

Fig. 6 shows the pertinent relative bandwidth  $\Delta E/E$ . It varies from well below  $10^{-3}$  at low, to roughly  $7 \times 10^{-3}$  at high photon energies. If needed in the chemical identification, the bandwidth can be reduced by going to higher reflection orders and/or using Si instead of Ge as monochromator material.

It may be noted from figs. 4-6 that a highly sophisticated source like the ESRF, when used with the described method of CT, is, although distinctly better with respect to speed and resolution, not overwhelmingly more favorable than "ordinary" sources like DORIS and SPEAR.

5. Experiment

In a preliminary experiment designed to test chemical sensitivity, we have investigated a model sample consisting of an Al foil 100  $\mu\text{m}$  thick with vacuum deposited layers of Cu and Ni. Layers were 2.5-3  $\mu\text{m}$  thick and had web-shaped interruptions due to shadowcasting during vacuum deposition caused by irregularly structured wire webs in front of the Al foil. Wires were about 60  $\mu\text{m}$  thick. Fig. 7 shows radiographs taken a few eV above and below the Cu and Ni K-edges. This sequence shows clearly the separate web structures of Cu and Ni although the atomic number difference is only one and respective material thicknesses are no more than a few  $\mu\text{m}$ . From the strong radiographic contrast, we conclude that at least 10% of the present amount of materials can be distinguished. Fig. 7 was obtained with DORIS running at about 5.3 GeV and 25 mA. Kodak Technical Pan 2415 film was used with exposure times running from 1 to 20 s.

6. Summary

We have shown that a very fast tomography method, capable of distinguishing neighboring elements in the periodic table at low concentration with spatial resolution of the order of 1-10  $\mu\text{m}$  is possible, by a combination of the following achievements of modern technology: SR, perfect crystal optics, high resolution two-dimensional detectors and microcomputers. We have calculated and compared the expected performance of six typical SR sources including one highly sophisticated source with respect to the new method of CT. Some conclusions regarding chemical sensitivity could be drawn from a preliminary experiment involving qualitative photographic measurements.

IV(a). LITHOGRAPHY/MICROSCOPY/TOMOGRAPHY

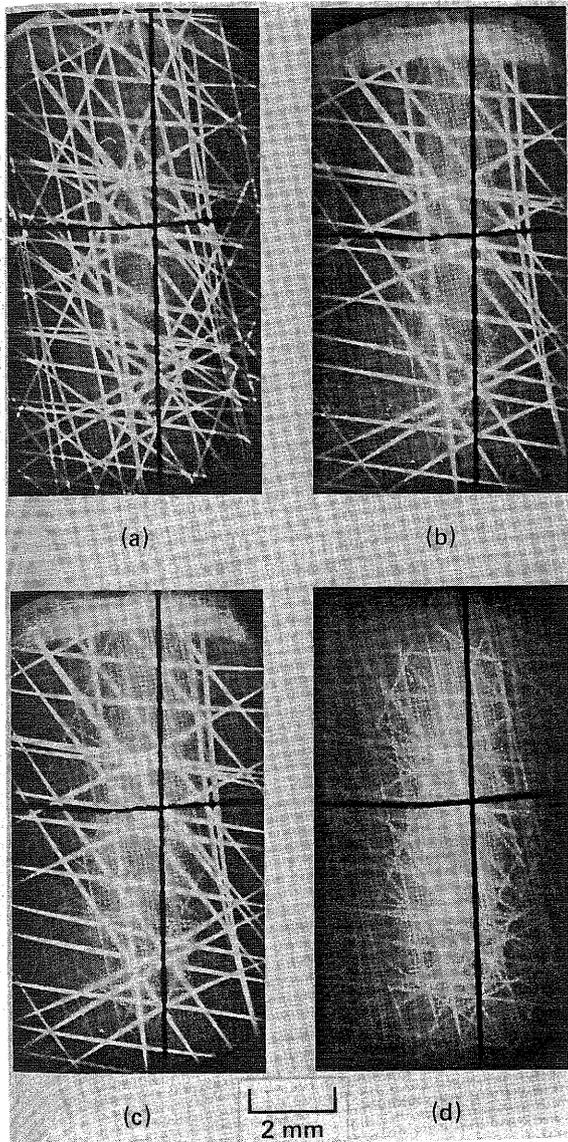
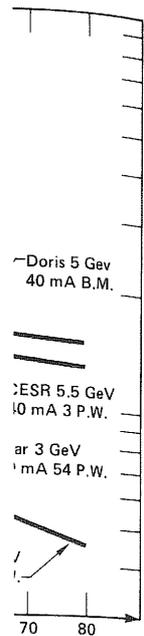
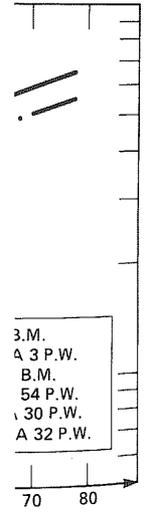


Fig. 7. (a) Web structured gaps in Cu + Ni Layers imaged at 23 eV above Cu K-edge, (b) 18 eV below Cu K-edge, (c) 26 eV above Ni K-edge, and (d) 22 eV below Ni K-edge.



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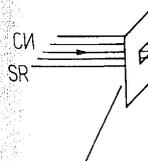
## SCANNING X- SYNCHROTRON

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Fig. 1. Layout