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HIGH RESOLUTION TOMOGRAPHY WITH CHEMICAL SPECIFICITY

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HIGH RESPLITION TOMOGRAPHY WITH CHEMICAL SPECIFICITY

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A very fast method of computerized critical absorption tomography featuring $\sim 10~\mu m$ spatial resolution and high chemical sensitivity is described. Synchrotron radiation is used and the method is especially suited to investigating small samples. From a preliminar, experiment it is found that layers of neighboring elements only .2 μm thick can be distinguished at medium atomic numbers.

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 non-medical 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 (SP) would be best suited to CT of small samples.

DISTRIBUTION OF THIS DOLLARS ON BUILDING

Because SP is verifient, very intense and easily turatle. CT entires SP can be shown to be superior to CT with conventional x-make, 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 ∞ one micron. 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 explosures. For example, at a source like BORIS operated at 3 GeV. 100 mA, bending magnet radiation, a more specimen of 3 mm diameter and 25 mm length would require about six minutes to image at a 25 micron cube voxel size.

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 degrees. 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 a 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.

- 2 -

Fig. 2 illustrates how sequential λ - and z-scat intentity measurements may be replaced by on simultaneous measurement. A wide bear of sufficiently small divergence combined with a two-dimensional array of charpe 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 bear with the required properties is obtained by combining an asympetric with a symmetric Bragg reflection as shown in fig. 2. The asympetry of the first reflection matches beam height to the height of the sample and at the same time reduces 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 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 um.

Presently two-dimensional CCD detectors with pixel size 10 X 10 µm 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.

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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 mm² at roughly 10^{-3} relative bandwidth. Thus, for a 10 X 10 µm pixel, photon fluxes of 10^{6} per second

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are not unrealistic. Assumine 1 t 57 detection efficiency, we have 10^4 to 5 ± 10^4 courts's pixel. In this case m = 3 m /HC in = 300 so measurements at 300 different angular settings are required. Assuming 18 statistical error we need 300 seconds measuring time for one energy, or 10 minutes for two energies. Allowing an additional 300 seconds for performing the 300 angular settings per energy, the sample may be imaged at 10 um resolution within 20 minutes. It should be noted that the huge number (2.25 ± 10^8) of individual intensity measurements are performed in 20 minutes, that the sample is imaged at as mary 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 imits of the method.

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

- 4 -

and thus the normal plur will necessarily increase, a compromise must be made between blur 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 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.

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* mn^2 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 currents from 40 to 100 mA. Some are bending magnet (BM) and others n-pole wiggler (nPW) 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

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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 to almost 10⁴. 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 µm 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 $7*10^{-3}$ 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 to 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 "ordinar," sources like DORIS and SPEAR.

EXPERIMENT

In a preliminary experiment designed to test chemical sensitivity, we have investigated a model sample consisting of an Al foil 100 µm thick with

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vacuum deposited layers of Cu and Ni. Layers were 2.5 to 3 µm 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 µm 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 µm. 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 seconds.

SUMMARY

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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 to 10 µm 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.

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REFERENCES

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- [1] R. A. Brooks and G. Di Chiro, Phys. Med. Biol. 24 (1976) 689.
- [2] P. Reimers, J. Goebbels, H. P. Weise and K. Wilding, Nucl. Instr. and Meth. 221 (1984) 201.
- [3] P. Burstein, P. J. Bjorkholm, R. C. Chase andF. H. Seguin, Nucl. Instr. and Meth. 221 (1984) 207.
- [4] M. Onoe, J. W. Tsao, H. Yamada, H. Nakamura,
 J. Kogure, H. Kawamura and M. Yoshimatsu, Nucl. Instr. and Meth. 221 (1984) 213.
- [5] L. Grodzins, Nucl. Instr. and Meth. 206 (1983) 541.
- [6] L. Grodzins, Nucl. Instr. and Meth. 206 (1983) 547.
- [7] R. W. James "The Optical Principles of the Diffraction of X-Rays," G. Bell and Sohns Ltd., London (1967).
- [8] N. M. Ceglio (1985), personal communication.

LIST OF FIGURES

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-- Fig. 1 Geometry for Ordinary Computerized Tomography

Fig. 2 Geometry for Fast, High Resolution Tomography with SP.

Fig. 3 Geometry for High Resolution Tomography which Minimizes Undesired Radiation

Fig. 4 Calculated Fluxes for Six Synchrotron Radiation Sources

Fig. 5 Spatial Resolution for Six Synchrotron Radiation Sources

Fig. 6 Relative Bandwidth for Six Synchrotron Radiation Sources

Fig. 7a Web Structured Gaps in Cu + Ni Layers Imaged at 23 eV above Cu K-edge

Fig. 7b 18 eV below Cu K-edge

Fig. 7c 26 eV above Ni K-edge

Fig. 7d 22 eV below Ni K-edge

-9-

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