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X-RAY AND ELECTRON RADIOGRAPHY OF THIN SPECIMENS

by

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ABSTRACT

Two radiographic methods are discussed for the examination of thin, low-density structures, using (1) low-energy X rays, and (2) high-energy electrons. The established techniques of X radiography are compared with the newly-developed techniques of electron radiography; the similarities and differences of the resulting images are noted together with those of visible light inspection. (auth)

I. INTRODUCTION

The principles of radiography have been applied for many years to the study of thin structures in biology and metallurgy. The advantage over ordinary photographic methods is that components which are nearly identical in light absorption properties (even opaque) may be differentiated in the radiograph because of their dissimilar X-ray and electron absorption properties. This fact often outweighs the main limitation of the radiographic technique, namely, low resolution. Where high resolution is required, X-ray microscopy, as developed by P. H. Kirkpatrick at Stanford¹, or electron microscopy--differing from the methods discussed in this paper by providing primary magnification through a focusing action--may be employed.

II. X RADIOGRAPHY

A. Technique

In X radiography, X rays from a tube with a small focal spot target, excited at low voltage (5 to 25 peak kilovolts), are incident on the specimen and fine-grain emulsion which are in intimate contact. The X rays traverse the specimen in relative amounts depending upon its density, thickness, and especially its effective atomic number, producing a latent image on the emulsion which is developed routinely in the darkroom.

B. Resolution

The obtainable radiographic resolution depends upon three factors: (1) the emulsion grain size, (2) the amount of diffusion caused by scattered radiation, and (3) the focal spot size and target-specimen-emulsion geometry.

1. In general, the limitation due to grain size is the most serious. The finest-grained emulsions manufactured at present are the Gaevert-Lippman and Eastman 649-GH and 548-O types.² The 649-GH plates are capable of resolving lines spaced one micron apart. This permits a magnification of one hundred diameters before an image spacing of 0.1 mm is reached, which is the widely accepted value of the minimum resolvable distance of the unaided eye when viewed at a normal distance of 25 cm. One can appreciate the inherent limitation of the radiographic technique with regard to resolution, by comparing this value with the maximum permissible magnification, on this standard, of the light microscope of two thousand diameters.

2. The magnitude and influence of scattered radiation are unimportant in low-energy radiography. As the X rays pass through matter they are diminished in intensity, or deviated in direction by electron interactions. The proportion of X ray quanta deviated, or scattered, to those absorbed is small; also, the scattered quanta have a high probability of being absorbed before leaving the material. Some fraction of the energy of a quantum which ejects a photoelectron may reappear, as the excited atom returns to its stable state, in the form of a quantum of low-energy fluorescent radiation, or other electrons called Auger electrons, which are readily absorbed. Thus the primary radiation activating the emulsion is accompanied by a small amount of X-ray and secondary electron emission which contribute a relatively slight diffusion to the primary image. (The background resulting from scattering

by intervening material, e.g., air, or by rays emanating from parts of the target surrounding the focal spot, is negligible.)

3. The limitation on resolution due to geometrical factors is usually less important than that due to the emulsion grain size. For thick specimens, or those in poor contact with the emulsion, the finite X-ray source produces a penumbra which exceeds the minimum resolvable distance limited by emulsion grain size. From simple considerations one can see that if two points in the specimen are separated by d , and their images just overlap at distance s from the points to the emulsion, then $d=rs$, where r is the ratio of the longest dimension of the projected focal spot to the distance between the focal spot and emulsion. Thus, a 2 mm diameter projected focal spot at 10 cm from the emulsion will permit a lack of object-emulsion contact of 50 microns before an inherent minimum resolvable distance of one micron is reached. Both factors can contribute simultaneously to the diffusion of the image. The geometrical limitation may be minimized by:

(a) reducing the distance between each point in the object and the emulsion, (b) decreasing the focal spot size, and (c) increasing the target-emulsion distance.

a. The specimen should be sectioned as thinly as conventional preparation equipment, e.g., microtome, will permit. Pressure should then be applied to provide uniform contact with the emulsion. This is best accomplished with a vacuum cassette³ (a Los Alamos model is shown in Figure 1) in which an opaque Wratten infra-red filter (No. 87) is forced, by atmospheric

pressure, into contact with the specimen and the sensitized surface. At extremely low operating voltages the cassette may have to be abandoned, and the exposure is then made in darkness in order to avoid filtration of the soft components and impractical exposure times. At still lower voltages the exposure must be made in vacuo.

b. The focal-spot size can be decreased by suitable electron optical design of the tube electrodes, but this is usually accomplished at the expense of the total permissible X-ray output. Focusing the electron beam at a minute target area places severe demands on the heat dissipation of the tube, necessitating a lowered tube current at the operating voltage desired. Special designs employing high-pressure fluid cooling, relative motion between target and focal spot, or a combination of both⁴ can be used to alleviate this difficulty.

In some tube designs, the electron beam can, in principle, be focused to a spot as small as 100 A in diameter. Enlarged shadowgraphs can then be made directly, at the sacrifice of short exposure times, by placing the specimen close to the spot and far from the emulsion.⁵

c. Increasing the target-emulsion distance presents practicable difficulties of space limitation, and, especially, an increase in required exposure time resulting from the $R^{-2}\exp(-\mu R)$ dependence of intensity (where R is the target-emulsion distance and μ is the air linear absorption coefficient).

Since the emulsion speed usually is an inverse function of its resolution, one ought to consider carefully the extent to which proposals (a), (b), and (c) should be applied, when a given recording medium is chosen, in order to retain practical limits on exposure time.

C. Contrast

1. General Considerations

The contrast in the radiographs is related intimately to the relative X-ray absorption properties and thickness of the components of the sample. Since the fractional transmitted intensity, I/I_0 , through a material of thickness, x , and linear absorption coefficient, μ , is governed approximately by the relation, $I/I_0 = \exp(-\mu x)$, one finds $\partial I/I = -x \partial \mu$.

In words, the radiographic contrast between two components of the same thickness, x , but of dissimilar average linear absorption coefficients, is proportional to the product of thickness times the difference between absorption coefficients, $\Delta\mu$. In order that $\Delta\mu$ be large, the operating voltage must be low (since μ for all components, and also $\Delta\mu$ between components, increases as the X-ray energy is lowered, except for absorption discontinuity effects).

The limitation on voltage is reached when I/I_0 is reduced to an excessively low value and then x must be decreased to compensate for this (as well as to increase resolution, as discussed in Section 3a above). This will lower the contrast, however. In general, both x and the operating voltage must be adjusted to

obtain the maximum contrast which is compatible with high resolution and reasonable exposure times.

2. Special Techniques

When working with materials in the medium or high atomic number range, one can increase $\Delta\mu$ and enhance the contrast considerably by homogenizing the radiation (e.g., using characteristic X rays) at an energy between those energy levels corresponding to the absorption discontinuities of the elements to be separated.⁶ Another method for increasing the absorption difference of biological components is to stain a particular tissue with a relatively high atomic number dye (e.g., lead acetate).⁷ Radiographic contrast will be discussed further in Section IV.

D. Required Materials

Considering the problems of obtaining high resolution, contrast, and reasonable exposure time, the requirements of the apparatus are as follows:

1. Ultra-fine-grain emulsion of good latitude, contrast, and sensitivity.
2. Means to provide good contact between specimen and sensitized surface.
3. Low-voltage, high-current, small projected focal spot X-ray tube.
4. Low inherent filtration.

III. ELECTRON RADIOGRAPHY

A. Technique

As described in Figure 2, electrons are ejected from a thin lead foil by X radiation excited by 250 peak kilovolts; the X rays are filtered to remove the soft components which might fog the film. The electrons then pass through a specimen in amounts inversely proportional to the number which are absorbed and scattered, forming a latent image on the photographic surface below.⁸

The principal advantage of electron radiography over thin specimen X radiography is that a conventional deep therapy medical or industrial type X-ray unit may be substituted for the special high-current low-voltage tube described above. Furthermore, electrons do not follow the same absorption laws as do X rays, and dissimilar radiographs are produced.

B. Resolution

The principal disadvantage of electron radiography is the characteristically lower resolution, by a factor of about four, obtained with respect to that of X radiography.

This low resolution, which permits acceptable magnifications up to only about 25 times when viewed at the normal distance, is due almost solely to the scattering of electrons in the emitter and specimen, and to the fact that electrons are emitted from the atom in nearly all directions with respect to the incident X-ray beam. These electrons, ejected by about 200 kilovolt X rays, are photoelectrons and, to a minor extent, Compton electrons. The presence of electrons is evidenced

by the fact that an intervening thin cellophane filter will reduce the film density to a small fraction of its unfiltered value..

From an elementary theory of scattering,⁹ one can calculate the average deflection due to the scattering of one megavolt electrons in only 25 microns of lead (the thinnest foil conveniently usable) as being more than one radian. The scattering angle is considerably greater for the lower electron energies encountered in electron radiography.

This is essentially why the resolution of electron radiographs is so low, and why it is so essential that the specimen be thin and in good contact with the foil and emulsion. A vacuum cassette is usually a necessity in this method of radiography.

C. Contrast

The absorption of a continuous spectrum of electrons (which is the energy distribution to be expected because of the continuous nature of the X radiation) in a material follows a curve shape similar to that of X-ray absorption with the important difference that a maximum electronic range exists, which is an increasing function of energy. It was found that the contrast between components is somewhat improved as the operating X-ray voltage is lowered, provided the specimen is thin enough to permit adequate electron penetration. Theoretical electron range values of electrons at several energies through air, water, (similar to soft biological tissue), and lead are stated in Table I, which is an interpolation of data given by Heitler.¹⁰ The empirical value would be on the order of 60 to 80 percent as great since the calculated range represents an integration over a tortuous path.

TABLE I

TOTAL PATH LENGTH OF ELECTRONS IN MILLIMETERS (THEORETICAL)			
Energy	Air (NTP)	Water	Lead
100 KV	150	0.15	0.03
200 KV	450	0.5	0.10
500 KV	1600	1.8	0.35

It was found by experiment, that the best radiographs, from the standpoint of contrast, resolution, and penetration of specimen were obtained in the operating X-ray range from 150 to 250 peak kilovolts. The higher voltage permits good penetrability and reasonable exposure times of several minutes. The lower voltage provides good contrast with ultra-thin specimens at the expense of longer exposure times. Filtration of about 5 mm copper plus 5 mm aluminum (to absorb characteristic copper radiation and Compton scatter) is considered adequate. Greater filtration increases exposure times unnecessarily--lesser filtration begins to diminish contrast by increasing the background radiation density. Below 150 peak kilovolts long exposure times are required and sufficient penetration is often impossible to obtain. It is likewise impractical to operate much above 250 peak kilovolts; for example, electron radiographs taken at 1000 peak kilovolts are very diffused and show significant background density due to excessive scattering.

The Eastman 649-GH spectroscopic plates were found to provide excellent contrast, with reasonable exposures. They were used throughout the experiments, although their high resolution was not considered to be essential.

Electron radiography with beta-emitting isotopes has been used successfully.¹¹ No elaborate equipment is required and the background radiation density can be considerably reduced, but exposure times required are often excessively long.

IV. COMPARISONS

In Figure 3, prints of an X radiograph, electron radiograph, and transmission photomacrograph of a section of rat tooth 25 microns thick are shown together for comparison. Figure 3A is a positive reproduction

of an Eastman 649-GH plate exposed by X rays emanating from a tungsten-target, 1.5 mm projected-square focal spot tube operated at 8 peak kilovolts and 380 milliamperes-minutes at 25 cm from the plate. The X rays were filtered by a one millimeter beryllium window, the air, and a Wratten 87 gelatin filter. The plate reproduced in Figure 3B was made essentially by electrons ejected from a 25 micron lead foil with X rays from a tungsten-target tube, 30 cm distant, excited at 250 peak kilovolts. A sheet vinylite-covered vacuum cassette was employed, and exposure was 60 milliamperes-minutes with the copper-aluminum filtration described previously. Figure 3C is a transmission photomacrograph print of the same specimen.

In Figure 4, the enlarged center portions of the first two prints are shown. Calcified tissue surrounds the dark inner regions of decalcified material. Both X rays and electrons pass through the latter with ease. The three black spots seen clearly in the center of the electron radiograph are holes in the specimen.

Since the X-ray absorption coefficient increases with about the fourth power of atomic number (except at absorption discontinuities), there is high contrast sensitivity between components in the X radiograph which differ in atomic number (e.g., calcified and decalcified areas) with little differentiation of density differences within both high and low atomic number regions.

The energy lost by moving electrons is directly proportional to the electron density in the matter traversed: $\rho N_0 Z/A$, where ρ is the physical density, N_0 Avogadro's number, Z atomic number, and A atomic weight. $N_0 Z/A$ is fairly constant for most elements; therefore, the electron absorption is approximately proportional to physical density. Thus, there are density gradations in the electron radiograph, corresponding to

components differing in physical density regardless of atomic number. The contrast between the calcified and decalcified areas (large atomic number differences) in the electron radiograph reproduction is not as pronounced as in the X radiograph, but the density variations in the central region are sharply evident (note the difference between the holes and the surroundings--where holes are interpreted in terms of density changes from tissue to air).

The photomicrograph, of good resolution, reveals differences in light absorption very well but no contrast between different elements of similar light absorption properties.

The differences in the absorption characteristics of these processes are further illustrated by Figure 5, in which reproductions of an electron radiograph and a transmission photomacrograph are shown of the same piece of typed bond paper. In contrast to the previous example, the electrons, sensitive to physical density changes, do not differentiate the typing, regardless of which side of the paper faces the emulsion; the light, of course, does. An X radiograph, not shown, separates the paper and typing, for apparently the average atomic number of the typing differs somewhat from that of the paper.

Another comparison between the radiographic and photographic methods is shown in Figure 6. The specimen is a 125 micron section of a baby mouse skull which contains a bone deposition of plutonium by previous injection of a salt of the metal into the animal. The radiographs and photomacrograph are taken by the same techniques as those of Figure 4 except that the X radiograph was exposed at 12 peak kilovolts (the higher voltage was necessitated by the increased specimen thickness) and 40 milliamperes-minutes. Again, the radiographs show higher contrast between the bone and other tissue than does the photomacrograph. The electron radiograph

is by far the inferior of the reproductions because of the excessive thickness of the specimen. As previously discussed, electron scattering requires that the sample be ultra-thin. In this case, however, it is five times thicker than the rat tooth section of Figures 4 and 5.

A comparison between X radiography, electron radiography, and photography can best be made by reference to Table II.

TABLE II

COMPARISON OF METHODS			
	X Radiography	Electron Radiography	Photography
Radiation medium used	5-25 Kilovolt X rays	100 - 200 kilovolt electrons	visible light (or ultra-violet light)
Contrast obtained	Difference in atomic numbers	Difference in physical density	Difference in light transmission
Maximum permissible magnification	100 x	25 x	2000 x
Apparatus required	Low-voltage, high-current, small focal spot X-ray unit with low-absorption window (and vacuum cassette)	150-250 peak kilovolt X-ray unit (and vacuum cassette)	Photomicrographic equipment
Advantages	Simple interpretation of specimen components (without staining) with three dimensional view.		High resolution

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Figure 1

Vacuum cassette with plate
and specimen in place.

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Figure 2

Procedure for making electron radiographs.

FIGURE 3
RAT TOOTH SECTION 25 MICRONS THICK
(A) X RADIOGRAPH
(B) ELECTRON RADIOGRAPH
(C) PHOTOMACROGRAPH

FIGURE 4

RAT TOOTH SECTION CENTER PORTION

(A) X RADIOGRAPH

(B) ELECTRON RADIOGRAPH

FIGURE 5
IDENTICAL PIECE OF TYPED BOND PAPER TAKEN BY
(A) VISIBLE LIGHT AND
(B) ELECTRONS.

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FIGURE 6
BABY MOUSE SKULL 125 MICRONS THICK
(A) X RADIOGRAPH
(B) ELECTRON RADIOGRAPH
(C) PHOTOMACROGRAPH