COHERENT X-RAY IMAGING FOR CORROSION EVALUATION: A PRELIMINARY ASSESSMENT

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INTRODUCTION

Corrosion products sometimes complicate the task of measuring metal loss. For example, in layered joints in aircraft, the products of corrosion usually remain trapped between layers. Their presence interferes with ultrasonic and x-ray measurements. Recently, Compton backscatter tomography has been suggested as a means of measuring the amount of metal loss resulting from corrosion. This x-ray method is appealing in that it is one-sided. However, no successful results seem to be yet reported. Suggested reasons for this have included long counting times [1] and poor contrast between metal and corrosion products [2]. Both these problems are related to the high resolution required.

Coherent x-ray imaging could circumvent the spatial resolution problem. It is much like dark field imaging in the electron microscope. Just as the microscopist can "light up" a particular phase such as a precipitate, so can a particular phase be selected for x-ray imaging. In both cases diffraction from the crystal lattice is the key to isolating the phase. Furthermore, because the strength of the diffracted beam is proportional to the volume of the diffracting phase, it is possible to generate a display in which intensity is proportional the amount of crystalline metal in a volume element. The size of the element may be made as large as desired.

COMPTON BACKSCATTER

Evaluation of corrosion losses by Compton backscatter is done by imaging a volume to be examined and then measuring the amount of metal in the image. The task can be divided into two parts: distinguishing metal from non-metal and then the measurement itself. The first part depends on the counting statistics; the second part depends on the spatial resolution. The overall passage of a scattered ray is ade-

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quately described for aluminum at energies near 150 KeV by adding an absorbtion term to the Klein-Nishna formula. The incident leg of the ray is the incident beam. A ray pencil within the scattered beam forms the second leg. For the sake of compactness, beam softening as a result of scattering will be treated by averaging the extinction coefficient. The Klein-Nishna formula is given in standard texts [3].

$$\frac{\Psi_{out}}{\Psi_{in}} \cong \frac{r_0^2}{2} Z N \left(\frac{\nu'}{\nu_0}\right)^2 \left(\frac{\nu_0}{\nu'} + \frac{\nu'}{\nu_0} - \sin^2 2\theta\right) \Omega t e^{-2\bar{\mu}l} \qquad (1)$$

Here, Ψ_{in} is the incident flux, Ψ_{out} is the received flux, r_0^2 is the classical electron radius squared, 7.95E-30 m^2, Z is the atomic number, N is the number of atoms per cubic centimeter, ν' is the frequency of the scattered photon, ν_0 is the frequency of the incident photon, 2θ is the scattering angle, Ω is the detector solid angle, t is the scattering path length within the volume element being considered, $\bar{\mu}$ is the average extinction coefficient over the ray path, and l is the path length.

Inspection of equation (1) shows that scattering is proportional to the NZ product which is the electron density. Equation (1) may be rewritten using aluminum as a reference. For 125 KeV. photons scattered through a 135 degree angle,

$$\frac{\Psi_{out}}{\Psi_{in}} \cong 0.4\ \Omega t \frac{\chi}{\chi_{Al}} e^{-2\bar{\mu}l} \qquad (2)$$

where χ is the electron density in the scattering material and χ_{Al} is the electron density in aluminum. Clearly, apart from contrast degradation within the imaging system, the contrast ratio between voxels is simply their electron density ratio. The electron density for pure aluminum may be calculated while effective values for other materials may be obtained from x-ray attenuation data. Here are some values for comparison.

Table 1, Effective Electron Densities

Pure Aluminum	4.76E29/m^3
Fuselage Skin Sample (2024)	6 E29
Corrosion Product Sample (from Al)	2-3 E29

The fuselage skin and the corrosion product values are both from attenuation data uncorrected for the photoelectric effect and are only approximate. They suggest a contrast ratio of about 2:1 between aluminum sheet metal and its corrosion products. Measurements made of a corrosion phantom using the Philips Com-Scan system (courtesy of North American Philips, Norcross, GA) at 160 KVP. appear to be consistent with this estimate.

Noise represents uncertainty in the value of the signal. In order to distinguish between corrosion and metal, the inherent contrast ratio must not be degraded to such an extent that it falls below the threshold of recognition. In backscatter imaging systems, shot noise is the major limita-

tion. Thus we may establish a criterion based on the idea that shot noise will not reduce, for example, the 2:1 contrast ratio given above, below a threshold level of a human observer, 1.03:1 [4]. Shot noise results from the Poisson distribution of received photons in a scattering process. The Poisson distribution is itself a limiting case of the binomial distribution where the probability is very small but the sample is large. Since there exists a standard transformation between the binomial and the normal distributions, a normal approximation will be used in much of the following. However, a normal distribution is symmetric and a Poisson distribution is not. The approximation can only be expected to hold near the mean. Given this caveat, the criterion may be written using equation A1,

$$1-c_t > \frac{n_{corr}}{n_{Al}} + \varepsilon \left(\frac{n_{corr}^2}{n_{Al}^3} + \frac{n_{corr}}{n_{Al}^2} \right)^{\frac{1}{2}} \tag{3}$$

where c_t is the threshold contrast, N_{corr} is the number of counts detected for the non-metal region voxel, N_{Al} is the number of counts for the metal region voxel and ε is a constant amounting to the number of standard deviations required for a given certainty. This formula presupposes neither a normal nor a symmetric distribution. However, it is based on a series expansion which neglects higher order terms. Since the ratio of N_{corr} to N_{Al} is the electron density ratio, this relation may be solved. Using a normal approximation and the values in Table 1, for fuselage skin, 95% of the time, there will be at least a 5% contrast between metal and corrosion product so long as there are at least 14 counts in each voxel.

To begin calculation of the required counting time, consider the sampling requirement. Each voxel represents a sample of the object space. Because the phase relationship between a boundary in the object space and its representation in the image space is unknown, the spatial sampling frequency must be at least twice the required resolution. For a "pinhole" camera, usually represented by a slit, the geometric resolution, the inverse of the maximum sampling frequency, is approximated by (see [5] for example),

$$r \approx 2\,d \left(1 + \frac{l_1}{l_2} \right) \tag{4}$$

where r is the smallest well-resolved dimension, d is the aperture, l_1 is the distance between the aperture and the object, and l_2 is the distance between the aperture and the imaging plane. When this last dimension is relatively large, the resolution becomes twice the aperture width. The solid angle subtended by the detector is that subtended by the aperture. If one wished to resolve metal loss to 0.005", the aperture would be only 0.00125" wide. Usually systems are configured so that the aperture need only resolve in one dimension. In that case a slit could be used. A slit 0.00125" by 1" placed 0.25" from the volume element being examined would subtend a solid angle of approximately 0.005 sr. If that element were 0.1" below the surface of an aluminum structure, then by equation (2) the ratio of re-

ceived flux to incident flux through the element would be 1E-7. The counting time per volume element is that required to get the number of counts given by equation (3), 14 counts in the example. The range of possible incident fluxes varies considerably. In the example, counting times between 0.5 and 700 seconds per voxel are expected.

COHERENT IMAGING

In order to measure by backscatter the amount of metal lost due to corrosion, a large number of readings must be taken, nearly four million per cubic centimeter in the example given. Alternatively, imaging only the lattice allows the intensity of each voxel to be proportional to the amount of metal therein. The spatial resolution may be decreased even to the point where the thickness of the volume element is equal to the thickness of the structure being examined. And, because imaging is from peaks in the scattering cross-section, the received signal is higher. The height of a peak may be many times the background level; even where the detector collects the entire peak at once, the received signal for (331) and (420) peaks in aluminum using filtered Mo K-alpha radiation have been found to be three to seven times higher than background where only a fraction of background is due to Compton. On the other hand, coherent imaging is complicated by noise due to grain size and textural variations. Grain size noise limits spatial resolution. The significance of textural noise has yet to be evaluated. Coherent imaging is an outgrowth of techniques used to measure the volume fraction of phases by diffractometry. Five to ten per cent represents the limit of accuracy in such measurements using conventional diffractometers [2].

Analogous to equation (1) it is possible to write an equation describing the coherent imaging process. The ratio of received to incident flux is given by,

$$\frac{\Psi_{out}}{\Psi_{in}} = t_f\,(1+\alpha)\,r_0^2 \cdot P \cdot FF^* \cdot L_p \frac{\lambda^3\, m_{hkl}}{V_c^2\, 32\pi}\, t \cdot \frac{s}{R} e^{-2\bar{\mu}l} \qquad (5)$$

where t_f is a texture factor, α is a small correction for thermal diffuse scattering [6], P is the polarization factor, FF^* is the square of the magnatude of the structure factor, L_p is the Lorentz factor for powder diffraction, is the wavelength in meters, V_c is the volume of the unit cell in m^3, m_{hkl} is the multiplicity factor, s is the length of the detector aperture in the same units as R, the effective distance between the detector aperture and the voxel. This expression is obtained by regrouping the terms of an expression given in Appendix C of the text by Schwartz and Cohen [7]. The two geometric factors in (5) are,

$$P = \frac{1+\cos^2 2\theta}{2} \qquad (6)$$

$$L_p = \frac{1}{\sin^2\theta \cdot \cos\theta} \qquad (7)$$

Note that the scattering angle is two theta as conventionally defined for diffraction and for Compton scattering above. The solid angle of the detector does not appear explicitly because the scattered parallel beam takes the form of hollow

cones in which the "wall thichness" is constant with R. Through the texture factor, there is a variation in (5) with rotation around the cone axis, the delta-angle in diffractometry.

As with Compton backscatter imaging, noise determines the accuracy of measurement. The major contributions to noise come from counting variability and variability in the number of reflecting grains due to textural variation or simply due to probability. This last source is termed grain size noise while that due to texture is textural noise. Grain size noise can be calculated from the grain size. It decreases as the beam gets larger because more grains are sampled. Textural noise results from changes in the orientation of grains as a function of position. In equation (5) it appears as variation in the texture factor. The effects of texture variability can be minimized by making s large and by summing several reflections such as the (331) and (420) used in the present work.

If the voxel thickness is set equal to the thickness of the structure being examined, a two-dimensional representation results. It is then possible to compare the metal thickness in one region with that in another reference region. To do this, both direct transmission and diffraction data are needed for each voxel. If the transmission measurement is made normal to the surface and both incident and diffracted rays make equal angles with the surface normal,

$$\frac{t_1}{t_2} = \frac{\sum_{hkl} n_{1,hkl} \cdot w_{hkl}}{\sum_{hkl} n_{2,hkl} \cdot w_{hkl}} \left(\frac{n_{2,t}}{n_{2,t}} \right)^{\frac{1}{\cos\theta}} \tag{8}$$

where t_1 is the thickness of metal at some point, t_2 is the thickness of metal at the reference point, $n_{i,hkl}$ is the number of counts in the ith measurement of the hkl reflection, w_{hkl} is the weight for the hkl reflection and $n_{i,t}$ is the number of counts in the ith transmission measurement. Using the approximations given in the Appendix, the variance of (8) is,

$$\sigma_R^2 = \left(\frac{n_{2,t}}{n_{2,t}} \right)^{\frac{2}{\cos\theta}} \left(\frac{(\Sigma n_1 \cdot w)^2}{(\Sigma n_2 \cdot w)^3} + \frac{\Sigma n_1 \cdot w}{(\Sigma n_2 \cdot w)^2} \right) + \left(\frac{\Sigma n_1 \cdot w}{\Sigma n_2 \cdot w} \right)^2 \cos\theta^{-2} \left(\frac{n_{2,t}}{n_{1,t}} \right)^{\left(\frac{2}{\cos\theta} \cdot 2\right)} \times$$

$$\left(\frac{n_{2,t}^2}{n_{1,t}^3} + \frac{n_{2,t}}{n_{1,t}^2} \right) + \sigma_G^2 + \sigma_T^2 + \sigma_E^2 \tag{9}$$

where $\Sigma n_1 \cdot w$ designates the weighted sum over all used reflections, σ_G^2 is the variance due to grain size, σ_T^2 is the variance due to texture and σ_E^2 is the variance due to other causes.

The variance in the number of diffracting grains due to their size is equal to the number of diffracting grains and

may be obtained for a single reflection as,

$$n_g = \frac{d_b^2 \, t \, \Omega \, m_{hkl} \, t_f}{64 \, V_g \sin \theta} \tag{10}$$

where V_g is the volume of a grain and d_b is the diamater of the beam. This expression from the probability of a grain having a reflecting orientation as given in [7]. The variance in the thickness ratio due to grain size is thus,

$$\sigma_G^2 = \frac{2 \, t_1}{n_g \, t_2} \tag{11}$$

EXPERIMENTAL WORK

The above methodology has been tried in the laboratory to a limited extent. The thickness loss due to corrosion was measured experimentally in a sample in which an excess of corrosion product was added. Two very fine grained 50-mil aluminum sheets were riveted together. One of the sheets was corroded in a specific area. The relative transmittance between the corroded and the non-corroded areas was measured using Mo K-alpha radiation passed through a LiF monochrometer.

Counts on corroded area = 2486
Counts in non-corroded area = 3297

The reduced transmission in the corroded area was due to the corrosion product. The diffracted intensity for the unity-weighted sum of (331) and (420) peaks equalled,

Counts in corroded area = 16840
Counts in non-corroded area = 24612.

Using (8) a metal loss of 7% was obtained against a true loss of 6%. The expected uncertainty, the standard deviation, obtained from equation (9) was 2.8% excluding grain and textural terms.

A preliminary attempt at estimating the effect of grain size and texture was made using a coupon cut from the fuselage of a large commercial aircraft. A broad-range diffractometer scan showed a peak structure similar to type 2024 aluminum. (The sample was supplied by Northwest Airlines without analysis.) The grain size was found to be approximately 50 microns. The sum of both grain size noise and textural variation was was compared between two regions of the coupon separated by one inch using three scans in each region. The measurements were made in transmission using a beam diameter of 0.032". The results are:

Table 2 Analysis of Variance

Area	(331) counts	(420) counts	Total
1	8132	5327	13459
1	8287	4619	12906
1	7798	5068	13066
2	7277	3318	10595
2	7682	4146	11828
2	7881	4373	12254

Area 1 mean = 13144 s.d. = 232

Area 2 mean = 11559 s.d. = 703
Fractional difference between areas = 12%
Grand Average = 12351
Variance between samples = 3766754, d.f. = 1
Fractional standard deviation between samples = 15.7%
Variance within samples = 411656, d.f. = 4
F= 9.1 F(0.95)=7.7 so there is a significant difference.
But, the least significant difference is 1649 which is less than the difference between the means.

The fractional standard deviation in the number of grains due to grain size was calculated for the 0.032" beam diameter and 50-micron grain size using equation (10) and was found to be 19% which is somewhat greater than the 15.7% observed standard deviation between samples expressed as a fraction of the mean. In making the above calculation the number of diffracting grains was taken as the sum of the estimated value for each reflection and the texture factor was taken as unity. Unaccounted correlation between the positions of the (331) and (420) reflecting planes is expected to affect the result.

CONCLUSION

When a layered joint corrodes, the x-ray transmissivity may either increase due to thinning, decrease due to corrosion product accumulation or even remain the same. A framework has been layed out for the study of possible ways of measuring corrosion by means of x-ray scattering under circumstances where transmission measurements would be uncertian. Two modalities, Compton backscatter and coherent imaging have been compared. Preliminary work has shown that both techniques appear to be feasible, but statistics have been inconclusive due to limitation of the strength of the primary beam and the small solid angle of detection available in commercial equipment.

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APPENDIX

Formulas for the variance of a ratio and of a number raised to a power near unity used above are:

$$\sigma^2_{\left(\frac{a}{b}\right)} = \frac{a^2}{b^3} + \frac{a}{b^2} \qquad \text{For Poisson-Distributed Variates} \qquad \text{(A1)}$$

$$\sigma^2_{a^p} \approx p^2 \left(\langle a \rangle\right)^{(2p-2)} \cdot \sigma^2_a \qquad \text{(A2)}$$

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