A TECHNIQUE FOR THE NONDESTRUCTIVE DETECTION OF VOIDS
AND COMPOSITION ANOMALIES IN METAL MATRIX COMPOSITE
WIRES USING X OR γ RAYS

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INTRODUCTION

An initial study of a technique proposed for the nondestructive testing of metal matrix composites is the subject of this paper. These composites are manufactured in the form of approximately 1/2-mm-diameter "precursor" wires. Larger structures are fabricated by diffusion bonding of lay-ups. Reliable nondestructive quality control indicators of wire integrity have not yet been developed although a number of possibilities are being examined. Testing of the precursor wires is difficult because current manufacturing processes produce wires that may be entirely satisfactory but that vary in cross-sectional geometry, in surface properties, and sometimes in the amount of matrix material that is present. Techniques based on observations of wire resistance, surface emissivity, and sound emission signatures are difficult to interpret because of these characteristics. Wire imaging using x-ray or neutron techniques is also difficult because large lengths of wire must be examined with a resolution in the plane of the wire exceeding 50 line pairs per millimeter. It is difficult to obtain such resolution with techniques that don't use film; also, with these techniques, there is the added burden of a large (approximately $10^5$ pieces of information per centimeter of wire) amount of data that must be processed and automatically analyzed subsequently using some flaw detection algorithm. The technique investigated in this study uses x or γ rays (neutrons could also be used) but not in an imaging mode. The amount of data that must be processed is reduced by a factor of about $10^4$. Additionally, the possibility of very
simple flaw, no-flaw signal level criteria is presented so that only minimal data analysis is required.

Consider the situation depicted in Fig. 1. The wire is submerged in and drawn through a liquid bath that has precisely the same linear attenuation coefficient for the radiation being used as a nominally ideal or standard wire, averaged over its cross section. As shown in Fig. 1, the immersed wire is illuminated with x rays, and a detector is placed so that it receives radiation from an area corresponding to the complete cross section of the wire and from a length that is arbitrary but assumed in this study to be one wire diameter. Immediately adjacent to the first detector, a second balanced detector is used as a reference detector by monitoring x-ray transmission through only the bath material. The signals from the two detectors are compared. If the wire has a standard or ideal composition, both signals will be the same. However, if the wire is not standard, the first detector will give a different signal level than the reference detector. By placing tolerances on the permissible differences between the two signals, a simple method is available for indicating the departure of a wire from its standard composition. Note that the liquid bath automatically adjusts for changes in the wire cross-sectional area and geometry. Thus, the technique is sensitive only to changes in the wire's average composition but not to size or shape variations. In the remainder of the paper, we study theoretically and experimentally a number of questions associated with the implementation of this proposed technique.

![Diagram of composite wire composition detector](image)
ANALYSIS

The problem of detecting voids or composition anomalies in a composite wire comprised of a matrix material (M) and a fiber (f) is considered. The wire is drawn through a bath that has a linear attenuation coefficient equal to the perfect or standard wire, as shown schematically in Fig. 1. The analysis will be done using aperture theory as it is applied to the study of diagnostic radiographic examinations. The nomenclature used in the analysis is given in Fig. 2. A gas-filled, high pressure ionization chamber that has a relatively high resolution imaging capability is assumed to be the detector. The detector selection is dictated by stability, noise injection, and resolution requirements. A more detailed discussion of the detector selection process is presented in another section of this paper. In this analysis section, only statistical noise associated with the x-ray photons will be considered. Other noise sources will be investigated in the experimental portion of this work.

The target selected in this study was a length of wire equal to one nominal diameter; thus the target aperture is $A_T = D^2$. The image area or aperture at the detector is $A_i$ where

$$A_i = M_T^2 A_T + (M_T - 1)^2 A_{FS} + A_D$$  \(1\)

Fig. 2. Schematic diagram defining nomenclature for the analysis.
Here, \( M_T \) is the target magnification and is given by

\[
M_T = \frac{d_s + L + d_A}{d_s + (L/2)}
\]  

(2)

Because the field illuminated by the x rays is of limited extent and the target is thin, the effects of scattered radiation can be neglected. 6

The x-ray source will be specified by giving its output at an energy \( E \) and a distance of 100 cm as \( \dot{\Phi}_o(E) \) photons/cm² sec keV. For a unit energy interval the fluence rate at the target is thus \( \dot{\Phi}_o(E)(d_s + L/2)^{-2}10^4 \).

The primary photon fluence rate in the image plane is

\[
\dot{\Phi}_o(E)10^4 \exp[-\mu_{STD}(E)L]/(d_s + L/2)^2 M_T^2
\]

(3)

where \( \mu_{STD}(E) \) is the absorption coefficient of the bath and the standard wire. The number of photons in the image area \( A_1 \) for an exposure time \( \langle t \rangle \) is

\[
\langle t \rangle \frac{\dot{\Phi}_o(E)10^4 \exp[-\mu_{STD}(E)L]A_1}{(d_s + L/2)^2 M_T^2} = \phi_I(E) A_1
\]

(4)

For photons of energy \( E \) absorbed in a detector, the signal resulting from the absorption is, to a first approximation, proportional to the photon energy. Thus, the photons are weighted by their energy and by the absorption coefficient. The number of events that are recorded is \( \phi_I(E)A_1\eta(E) \), where \( \eta(E) \) is the detector absorption efficiency. The standard deviation of absorbed energy is thus \( [\eta(E)\phi_I(E)A_1]^{1/2} \Delta E \), where \( \Delta E \) is the difference in energy between the energy \( E \) and the nearest absorption edge. Contributions from K fluorescence and Auger electrons, which can be important (10 - 30%), are ignored and need not be considered at this point.

If radiation with a spectrum of energies is present, the sum over all energies becomes

\[
E_{\text{max}} \left[ \int_0^{E_{\text{max}}} \frac{\langle t \rangle \dot{\Phi}_o(E)10^4 A_1}{(d_s + L/2)^2 M_T^2} \exp[-\mu_{STD}(E)L] \eta(E)(\Delta E) dE \right]^{1/2}
\]

(5)
For a target of aperture $A_T$ the signal for photons of energy $E$ will be

$$\langle t \rangle \phi_o(E) \exp(-\mu_{STDL}) \int_{A_T} \Delta \mu(E) \ell \, dA_T \right] 10^4 \Delta E n(E)/(d_s + L/2)^2$$

(6)

where $\Delta \mu(E)$ is the difference in linear absorption coefficients between a wire and the standard wire. Following Reference 2, we will make the assumption that

$$A_T^{-1} \int_{A_T} \Delta \mu(E) \ell \, dA_T = \Delta \bar{\mu}(E) \ell_{\text{eff}}$$

with

$$A_T^{-1} \int_{A_T} \ell \, dA = \ell_{\text{eff}} = 0.7D$$

where $D$ is the wire diameter. Integrating over all photon energies results in

$$E_{\text{max}} \frac{\langle t \rangle \phi_o(E) \exp(-\mu_{STDL})L \Delta \bar{\mu}(E) \ell_{\text{eff}} 10^4 \Delta E n(E)(d_s + L/2)^{-2} \, dE}{E_{\text{max}} \frac{\langle t \rangle \phi_o(E) \exp(-\mu_{STDL})L \Delta \bar{\mu}(E) \ell_{\text{eff}} 10^4 \Delta E n(E)(d_s + L/2)^{-2} \, dE}$$

(7)

for the absorbed energy associated with the signal.

The signal to noise ratio (SNR) is

$$\text{SNR} = \frac{\int_0^{E_{\text{max}}} \langle t \rangle \phi_o(E) \exp\left(-\mu_{STDL}(E)L\right) \Delta \bar{\mu}(E) \ell_{\text{eff}} 10^4 (d_s + L/2)^{-2} \Delta E n(E) \, dE}{\int_0^{E_{\text{max}}} \phi_o(E) 10^4 (d_s + L/2)^{-2} \, dE}$$

(8)

In order to relate this expression to the normally measured output of an X-ray tube $\varepsilon_M$ in Roentgens per second (R/S), we have

$$\varepsilon_M = \frac{10^4}{d_M^2} \int_0^{E_{\text{max}}} \phi_o(E) \frac{(\mu_{en}/\rho)_{\text{AIR}}}{K} E(1.6 \times 10^{-9}) \, dE$$

(9)
where \( d_m \) is the distance at which \( \dot{\varepsilon}_m \) is measured. The energy absorption coefficient for air \( [(\mu_{en}/\rho)_{AIR}] \) was obtained from Reference 7. The form of \( \dot{\phi}_o(E) \), which has units of photons/sec cm\(^2\) keV, is assumed to be Krämer's expression,

\[
E\dot{\phi}_o(E) = \dot{C}(E_{\text{max}} - E)\exp\left[-\mu_{\text{INH}}(E)l_{\text{INH}}\right]
\]

Here, \( \mu_{\text{INH}}(E) \) will be assumed to be for aluminum and \( l_{\text{INH}} \) will depend on the x-ray generator. A typical value for \( l_{\text{INH}} \) may be 0.5 mm, so

\[
E\dot{\phi}_o(E) = C(E_{\text{max}} - E)\exp\left[-\mu_{\text{INH}}(E)0.05\right]
\]

and \( \dot{C} \), which has units of photons/cm\(^2\) sec keV, can be evaluated as

\[
\dot{C} = \frac{d^2_m}{10^4} \frac{E_{\text{max}}}{\varepsilon_M K} \int_{E_{\text{min}}}^{E_{\text{max}}} (E_{\text{max}} - E)\exp\left[-\mu_{\text{Al}}(E)0.05\right] (\mu_{en}/\rho)_{AIR}(1.6 \times 10^{-9})\,dE
\]

Here, \( K \) is the energy absorbed in 1 gm of air that represents 1 R (87 ergs, and 1 keV = 1.6 \times 10^{-9} \text{ ergs}).

It is of interest to estimate the current that the detector produces. If it is assumed that the area of one electrode segment is \( A_i \) and that approximately 0.024 keV is required for the creation of an electron-ion pair in krypton, \( 10 \) we get

\[
i = A_i \int_{E_{\text{min}}}^{E_{\text{max}}} \dot{\phi}_o(E)\exp\left[-\mu_{\text{STD}}(E)L\right] \frac{[-\mu_{\text{STD}}(E)L]}{(d_s + L + d_A)^2}
\]

\[
10^4(\Delta E/0.024)\eta(E)(1.6 \times 10^{-19})\,dE
\]

Also, the actual signal current (in amperes) is

\[
i_s = A_i \int_{E_{\text{min}}}^{E_{\text{max}}} \dot{\phi}_o(E)\exp\left[-\mu_{\text{STD}}(E)L\right] \frac{[-\mu_{\text{STD}}(E)L]}{(d_s + L + d_A)^2}
\]

\[
l_{\text{eff}}10^4(\Delta E/0.024)\eta(E)(1/6 \times 10^{-19})\,dE.
\]
It is necessary to determine $\Delta \mu(E)$, which requires some assumptions about the nature of the anomalies in the composite wires. After studying several sample wire cross sections, we found that in many cases the matrix material simply did not penetrate the fiber bundle. The fibers appear to be distributed independent of the presence or lack of matrix material. Thus, the anomalies appeared to be best approximated by a modeling that assumes an air void or simple lack of matrix material with the volume fractions $(X)$ related such that

$$X_M + X_V = X_{MSTD}$$

and

$$X_f = X_{fSTD} \hspace{1cm} (14)$$

The subscript STD represents the ideal or standard composite wire. For this situation, if $\mu_W(E)$ refers to a wire that is being tested,

$$\Delta \mu(E) = \mu_{STD} - \mu_W = X_V \mu_M(E) \hspace{1cm} (15)$$

Another possibility for modeling an anomalous wire is that the composition changes, but there are no voids. For this case

$$\Delta \mu(E) = \frac{\mu_M(E)(\rho_W - \rho_f)}{\rho_M - \rho_f} + \frac{\mu_f(E)(\rho_M - \rho_W)}{\rho_M - \rho_f} \hspace{1cm} (16)$$

Note that the standard wire is such that

$$\mu_{STD}(E) = \frac{\mu_M(E)(\rho_{STD} - \rho_f)}{\rho_M - \rho_f} + \frac{\mu_f(E)(\rho_M - \rho_{STD})}{\rho_M - \rho_f} \hspace{1cm} (17)$$

Still another possibility is that the wires, because of their construction, have a fixed amount of fiber but varying amounts of matrix material, along with possible voids. In this case there is no readily specified "standard" wire, and it is appropriate for the bath to have an absorption coefficient equal to the matrix material absorption coefficient. We use the same image area as in the previous analysis. (Actually we might want to look at slightly larger image areas to ensure that all of the wire material is always in view.) The detection task is to observe voids in a matrix material in the presence of the fibers. The transmission through the matrix
material with a volume fraction of fibers \( X_f \) (and \( X_M = 1 - X_f \)) is given by

\[
\bar{\mu}(E) = \mu_M(E)X_M + \mu_f(E)X_f
\]  

Here, the amount of matrix material or its equivalent bath material is determined by the bath dimension \( L \) and not simply by the nominal wire diameter. We will use superscript primes to denote this case for the volume fraction. The basic photon fluence that provides the noise is the same as in Eq. (3), except that \( \bar{\mu}(E) \) replaces \( \mu_{STD}(E) \) and Eq. (5) becomes

\[
E_{max}^{1/2} \left\{ \int_{0}^{E_{max}} \phi_o(E) \exp[-\bar{\mu}(E)L] \Delta \mu(E) L 10^{4} (d_s + L/2)^{-2} (\Delta E)^{2} \eta(E) dE \right\}
\]  

The signal is

\[
\int_{0}^{E_{max}} \phi_o(E) \exp[-\bar{\mu}(E)L] \Delta \bar{\mu}(E) L 10^{4} (d_s + L/2)^{-2} \Delta \eta(E) dE
\]  

The \( \Delta \bar{\mu}(E) \) can be determined from Eq. (14), with \( X \) appropriate to the present problem (i.e., based on both matrix and matrix equivalent materials.) Thus we have

\[
\Delta \bar{\mu}(E) = \mu_{v} \dot{X}_v
\]  

Again note that the \( \dot{X}_v \) is based on the bath depth and not on the wire diameter.

Other target models are possible and will be considered in a later paper if appropriate. Information on the nature of the defects might also be obtained by using two x-ray beams with different average energies.

CALCULATED RESULTS

Using the expressions developed in the preceding section, calculations have been made for SNR [Eq. (8)] and current [Eq. (13)] with the target model given by Eq. (21). The composite was assumed to be graphite fibers in an aluminum matrix. The wire void fraction \( X_v \) was assumed to be 0.01, the bath thickness 0.125 cm, and the nominal wire diameter 0.0625 cm. The detector, as represented by \( \eta(E) \) and \( \Delta(E) \), was assumed, for some of the calculations, to be a perfect detector with \( \eta(E) \) and \( E_K = 0 \). For other calculations, a
high pressure krypton or Freon detector was modeled by having \( n(E) = 1 \) but \( E_K = 14,300 \text{ keV} \). The carbon was assumed to have a density \( \rho = 2.00 \text{ gm/cm}^3 \), and the aluminum \( \rho_{\text{Al}} = 2.70 \text{ gm/cm}^3 \). The mass absorption coefficients used for carbon and aluminum were obtained from Reference 12. The discrete values from Reference 12 were fitted with polynomials for purposes of the computations.

The calculations were all done with the assumption that the output of the x-ray source resulted in a measured exposure of 1 R/sec at 100 cm. These values substituted into Eq. (12) provide a \( C \) for each kVp or \( E_{\text{max}} \) used in the calculations. The resultant values of SNR and current must be adjusted to account for the actual output of x-ray sources. A typical industrial x-ray source with a nominal 0.5-mm focal spot, no liquid cooling, tungsten target, and 0.5 mm of inherent filtration has an increasing output with increasing \( E_{\text{max}} \) (kVp), as shown in Fig. 3. This particular source will be used to convert the calculated SNR's and currents to predictions for a realistic composite wire quality control device.

With the conditions just described, we have calculated \( \text{SNR}/(R_{100})^{1/2} \) (note \( R_{100} \) \( \langle t \rangle = R_{100} \) ) and current, based on the source of Fig. 3, for a high pressure gas ionization detector. The results are shown in Figs. 4 and 5. Note that all the calculations are for an x-ray source to target distance of 10 cm. Other geometric parameters have been varied, with \( d_A \) going from 0.1 to 10 cm and the focal spot aperture \( A_{\text{FS}} \) taking values of 0.01 and 0.0225 cm².

![Tube output at 100 cm](image)

**Fig. 3.** Experimental output of Faxtron x-ray unit in Roentgen/sec at 100 cm.
Fig. 4. Calculated SNR per unit exposure of 1% void fraction in a composite wire.

Fig. 5. Calculated center electrode current of composite wire monitor for exposure to source in Fig. 3, but $d_s = 10$ cm.
The SNR results in Fig. 4 indicate that there is an optimum in SNR/R$_{100}$ at an $E_{\text{max}}$ of approximately 26 keV. However, the picture changes when the change of tube output with $E_{\text{max}}$ (Fig. 3) is taken into account. A typical electrode current curve is shown in Fig. 5, exhibiting a monotonic increase with voltage. SNR and signal current [Eq. (13b)] are shown in Fig. 6. The SNR monotonically increases with increasing $E_{\text{max}}$ to about 40 keV, after which there is a slight decrease up to the limit of the calculation at 45 keV. Conversely, the signal current increases throughout the voltage range considered. The usable lower limit to $E_{\text{max}}$ will be determined by system electronic noise or detector absorption efficiency, with system noise likely representing the most serious problem; however, this limit will depend on the details of the injected noise characteristics. This question will be discussed further in the section of this paper on experimental results. For our example, the signal current is of the order of $X_{\mu}L \approx 0.005$ for a 20-keV average photon energy and $X_{\mu} = 0.01$ times the total detector current. In the experimental section, the signal currents will be compared to observed detector system dark currents.

Fig. 6. SNR and signal current of composite wire monitor for exposure to source in Fig. 3, but $d_s = 10 \text{ cm}$. 
EXPERIMENTAL RESULTS

Experiments were conducted using the x-ray generator, whose output is shown in Fig. 3. The purpose of the experiments was, first, to establish the validity of the calculations as a significant system design tool and, second, to obtain preliminary measurements on selected composite wires.

The electrode configuration for the detector has been illustrated in Fig. 7. The bath and wire positioning system are shown in Fig. 8. To establish some confidence in the calculations, it is appropriate to compare calculated and experimental values of the electrode current and the balance energy $E_{\text{max}}$ for a known iodine concentration in the bath.

The bath liquid was composed of the commercial contrast agents Renografin 60 and 76. These liquids contain 0.290 and 0.370 gm/cm$^3$ of iodine, respectively. In addition, center electrode current measurements were made for a 1.25-mm-thick sheet of aluminum used on an attenuator.

The results compared to the expected values of center electrode current measurements versus tube voltage are shown in Fig. 9. The measured curve has roughly the predicted shape but is between 3 to 6 times below the prediction in the 20 to 35 kVp range. Some of this difference may be attributed to ion-electron recombination, some to incomplete absorption in the krypton, particularly at the higher energies. Finally, the alignment capabilities of the apparatus were limited and thus likely introduced inefficiencies. The exact causes for the low observed currents remains to be identified in the future. We do know by comparison

Fig. 7. Photograph of detector chamber and detector electrodes.
Fig. 8. Photograph of wire holder and bath.

to independent calculations that the predictions presented in this study are correct. It is important that the shape of the measured curves is reasonably in agreement with the prediction.

From measurements with the Renografin 60 and 76, as well as with the aluminum, effective attenuation coefficients based on the current measurements could be obtained for these substances. The results are shown in Fig. 10. They reveal considerable structure resulting from the presence of the K edge of krypton at about 14 keV and the distribution in the photon spectra after filtration by the absorbing material. The aluminum absorption coefficient generally drops more quickly with energy than either the 0.29 or 0.37 gm/cm³ iodine curves. The match energy for the 0.29 gm/cm³ iodine bath is about 22 keV; for the 0.37 gm/cm³ iodine curve, about 18.5 keV (by extrapolation of the results). Note that the two bath curves have quite similar shapes. The balance energies are achieved at smaller iodine concentrations than predicted in Fig. 6. This is not really surprising as the bath solution is not simply water and iodine but contains a number of other chemicals. The most significant result is that the bath behaves qualitatively in the manner that was predicted.

Some preliminary studies have been done on composite wires. A conventional film radiograph was taken of an aluminum wire, as well as "good" and "bad" composite wires. A set of microdensitometer traces of the film along an arbitrarily selected path perpendicular to the wires is shown in Fig. 11(a) and (b). In this figure, we present one set of traces with no bath fluid and another set with a
Fig. 9. Comparison of preliminary center electrode current measurements and predictions for experimental conditions.

Fig. 10. Measured effective absorption coefficients, using the center electrode current, of two baths with different iodine concentrations and aluminum.
Finally, detector dark currents were measured and found to be around $3 \times 10^{-12}$ A, comfortably below the predicted signal currents in Fig. 9 and even below the reduced levels that would correspond to the observation in Fig. 9. Detector noise injection does not appear to be a serious problem.
CONCLUSIONS

Both calculations and preliminary experiments demonstrate that the proposed technique for detecting voids and composition anomalies in precursor composite wires is feasible. Using commercially available industrial X-ray sources with stationary, uncooled anodes, void fractions in the 1 to 3% range should be detectable at linear wire speeds of around 10 cm/sec. The resolution along the wire length would be between 1/2 and 1 mm. Higher linear speed would be possible with reduced wire length resolution.

The bath immersion strategy appears to be a valid way of removing the uncertainties caused by geometric and physical property variations in graphite fiber, aluminum matrix composite wires.

ACKNOWLEDGMENT

The authors thank M. E. Brennan for her expert advice on the computations and R. Ruiz for his design work and assistance with the experiments. This work was sponsored by the Naval Surface Weapons Center under Contract No. F04701-81-C-0082.

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