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Quantitative Evaluation of Material Composition of Composites
Using X-ray Energy-dispersive NDE Technique

by

Ting, Jason

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Ames Laboratory, U.S. DOE

Iowa State University

Ames, Iowa 50011

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DEDICATION

To My Father and Mother
and My Sister Teresa.

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1. INTRODUCTION

1.1 Need for QNDE in composite studies

In a wide range of situations the need for characterization of material properties of man-made and natural materials arises. These situations can include inspection during fabrication of material, and in-service inspection of mechanical parts as they wear and near their design life. The need to characterize the material of interest involves all aspects of material performance, such as: the strength of the material, fracture toughness, density, hardness, material uniformity, and conductivity, etc. We present here a technique that is capable of quantitatively determining composition, density and material uniformity. It should be pointed out that no one technique can measure all properties of interest.

The development of new materials for commercial usage is brought about by increasing demand for high strength, high fracture toughness, light weight, and low cost of materials. Very few manufacturing processes can be standardized to guarantee the quality of the product. If these new materials are to meet the design requirements, some form of testing of the finished products will certainly be necessary. Therefore, the challenge to the field of nondestructive evaluation (NDE) is to expand beyond simple flaw detection into material characterization for the purpose of estimating mechanical and physical properties of the new materials. In this thesis, a new quantitative NDE x-ray technique is presented that meets this challenge. Nondestructive material characterization for material variations can be used to achieve higher reliability in the manufacturing processes, and also can lower life-cycle cost of the product with in-service NDE inspections. As performance demands for new advanced materials increase, the manufacturing quality control will become more stringent. Recently, material characterization for in-service inspection has become an important issue in the commercial aircraft industry. As

the world aircraft fleet is becoming older, the material characterization for the purpose of corrosion detection is extremely important in order to extend the design-life of older aircraft.

The advanced materials that are of interest in this study are composite materials. There are many types of composites used in structures. The most widely used are low-density composites consisting of high strength fibers, like glass and carbon, in combination with thermoset resins such as polyesters and epoxies. This is particularly true for aerospace and automobile industries. Many conventional alloys in the manufacturing of aircraft and automobiles and other items are increasingly being replaced by composite materials. The common composites used are metal-matrix composites and graphite-epoxy composites. This is mainly because the directional strength and the fracture toughness of the composite products can be tailored to match the required performances of the mechanical part for load-bearing purposes; while, at the same time, the composite products can be tailored to minimize the weight and cost of the material.

In NASA Aircraft Energy Efficiency (ACEE) program [14,20,22], which spanned the mid-70's and into the 80's, applications of advanced composites were demonstrated in secondary and non-load-bearing primary structures for commercial transport airplanes. Thus, as a result, as much as 5% of the structures in newer aircraft such as Boeing 757 and 767 and the Airbus 320 are being converted from conventional alloys to graphite-fiber-reinforced-epoxy composites. This is accompanied by weight saving of more than 20% for some airplane secondary structures such as 727 elevator and DC-10 rudder, and primary structures such as 737 horizontal stabilizer and DC-10 vertical fin [29]. More recently, aircraft such as the Lear Fan is made almost exclusively of composite materials- about 90% graphite-epoxy composite. Therefore, over the past decade and a half, extensive research and application development have produced high performance graphite-epoxy composites with demonstrated material confidence, and predictability.

As aircraft industry advances, the industry pushes the application-envelop of composite materials. More graphite-composites are beginning to replace metal alloys in primary load-bearing structures. Thus, there is a demand for more stringent quality control of the composite and for better understanding of the material characteristics of the product. The benefits of composite materials are sometimes offset as it is difficult to control the manufacturing quality of the composite products. Composite products, and particularly fiber-reinforced plastics, often contain defects as a result of normal manufacturing procedures. In the final composite product, it is common to find matrix-rich regions and matrix-starved regions and voids. These defects affect the mechanical properties of the composite products. For example, 1% material variation in graphite-epoxy composite product results in 3% loss of composite strength. It is common to find material variation of up to 10-15% in graphite-epoxy composite products. Therefore, quantitative NDE is necessary for manufacturing quality control as well as for in-service damage detection of composite products.

The application of composites is very broad. Similarly the definition of composite will be very broad in this thesis work. We define the term composite as any physical system that is composed of two or more components distinguishable at the macro-level. We will study three different composite systems in this thesis. They are: graphite-epoxy composite system, bone-Plexiglas composite system, and a composite system of a metal and its corrosion product. The emphasis of this thesis is with two-component composite-systems. Nevertheless composite-systems with more than two components can be handled in the same manner discussed herein.

1.2 Review of previous studies

For traditional nondestructive evaluation by x-ray, conventional radiography using x-ray films and image intensifiers may be useful in certain cases. These techniques can be used to identify voids and regions of substantially varying density in simple composites. It is known

that a monoenergetic beam can be used for composite material characterization [30], provided it is a two-component composite, the total thickness of the composite is known and that the thickness remains constant. An alternative method known as energy-dispersive x-ray is presented in this thesis to measure material composition avoiding the need to have a constant total sample thickness. The idea behind the x-ray energy-dispersive technique is based upon the fact that elements and compounds have characteristic x-ray attenuation as a function of x-ray energy is shown in Fig. 1.1, and, that the rule-of-mixture holds for x-ray absorption measurements. This technique permits us to measure material composition in materials having two or more components. The energies have to be chosen because of the energy dependence of the elemental linear attenuation coefficients. For photon energy below 100 keV there is an important contribution to the attenuation coefficient from the photoelectric process, which leads to a strong dependence of the attenuation coefficient for each element on the atomic number, Z . At much higher x-ray energy, up to approximately 10 MeV, Compton scattering dominates. This energy range gives sensitivity, not to atomic number, but to the density of the material [9].

Previous work [16] to measure quantitative resin contents in graphite-epoxy composite using a x-ray radiography film technique has demonstrated that the technique is inaccurate. Martin concluded that x-ray films have insufficient sensitivity to detect a small change in composite density. In addition to this, the x-ray attenuation coefficients of graphite and epoxy-resin are similar at all energies, so there is not enough contrast in the two material coefficients to detect the differences on films.

It has been suggested that measurements just made above and below the photoelectric absorption edge of a selected element would greatly enhance the contrast both in film-radiography [2,12] and in tomography [23]. Rudich et al. [25] developed a high flux x-ray tube for high output operation at 6 - 25 keV range for inspection of light elements such as graphite-epoxy composite. The inspection takes advantage of higher attenuation variations and

Figure 1.1. Linear attenuation coefficients of several elements

the photoelectric absorption K-edges, to increase the contrast and resolution to improve flaw detection. But the locations of the K-edge for carbon, aluminum and calcium are below 5 keV, thus such a technique is not feasible. In addition, care must be taken when working near the absorption edge of elements. Kerur et al. have shown that the rule-of-mixture breaks down when measurements were made at about 471 eV near the K-edge for chromium [8].

The main problem encountered when inspecting light elemental material such as graphite-epoxy composite is that the x-ray attenuation coefficients of graphite-fibers and epoxy-resin are similar, as both materials have same basic elemental composition of carbon polymers. Even then, single energy measurements by Shull et al. have shown promise in quantitative characterization of porosity in graphite-epoxy composites [26].

The energy-dispersive method has been used in clinical studies for measuring bone mineral and bone density [5,17,18,21]. All these studies were made with radioisotope x-ray sources. This restricted the choice of energies used in this energy-dispersive method. This problem can be addressed by the use of a x-ray generator and monochromator to select the desired energies. The use of a monochromator reduces the radiation dosage directly from the x-ray generator. Our study focus on the choices of x-ray energies by using a x-ray generator, but we will not address the use of a monochromator. Instead, the energy discrimination and resolution are resolved with the use of a germanium detector coupled to a multichannel analyzer.

1.3. Scope of the research

The purpose of this work is to develop a nondestructive evaluation technique by using x-rays that can quantitatively determine material composition, for material characterization, in composites. Such information can then be used to estimate the material properties such as the strength of the composite and the densities of the components.

The fact that elements and compounds have characteristic x-ray attenuation that depends on x-ray energy makes x-ray a natural tool for material characterization. Indeed, the advantages of this energy-dispersive technique over other x-ray detector systems such as films and fluoroscopy are its increased sensitivity to photon energy and photon intensity. The technique presented in this thesis work uses a germanium detector coupled to a multichannel analyzer that allows us to measure the photon intensities and photon counts with respect to energies. Therefore there is an abundant choice of energies for selection.

X-ray transmission methods can not distinguish the sequencing in which the components are ordered in a composite product. Thus, in this thesis, the word composite will not strictly refer to engineered structural composites such as graphite-epoxy composites or metal-matrix composites. We will restrict ourselves to studying two-component composites, and we will refer the word composite to any material that has two or more components.

Three composite samples studied in this thesis fall into three categories of general interest; they are: (1) a composite system of two materials with similar elemental compositions and material densities represented by graphite-epoxy composite, (2) a composite system of two materials with different elemental compositions and material densities represented by bone and Plexiglas, and (3) a composite system of two materials having approximately the same elemental compositions with different material densities represented by aluminum 2024 and its corrosion component, aluminum hydroxide.

The first composite system in this study is a composite of graphite-fibers and epoxy-resin matrix. Both materials have carbon as their main chemical constituent. Thus their x-ray linear attenuation coefficients are expected to be similar. The purpose of this study is to determine the sensitivity of this technique to measure the material variation in the composite samples for determining their fiber-to-resin ratios. Graphite-epoxy composites are increasingly replacing metal alloys of comparable strength, because graphite-epoxy composite is lighter than

metal alloys. Due to manufacturing variability graphite-epoxy composites often contain material variation in fiber-to-resin ratio. This defect affects the mechanical strength of the composite. This problem is addressed in this work and x-ray method is developed to measure the non-uniformity in composites.

The second composite system is a composite of bone and Plexiglas. We have used Plexiglas to simulate human tissue. Bone is essentially made of calcium phosphate and hydroxyapatite, while Plexiglas is made of acrylic; thus the x-ray attenuation coefficients and material densities are different for the two materials. The bone-Plexiglas composite samples are used to simulate osteoporosis in a human bone. Osteoporosis is a common bone disease in adults, where, the bone can lose as much as 40% density [21]. Osteoporosis usually results in loss of bone material, weakening the bone-strength. Although the osteoporosis usually takes months or years, early detection of bone loss can in many cases give information to avoid physical stresses that can result in bone fracture. The purpose of this study is to evaluate the sensitivity of this energy-dispersive technique in detecting the onset of osteoporosis by detecting the changes in bone density.

The third composite system is a composite of aluminum 2024 metal alloy and its corrosion product. The x-ray mass attenuation coefficients for the metal and its corrosion product are essentially the same, but the linear attenuation coefficients are different for the two components because their densities are different. Metal corrosion is of interest in this work, because metal corrosion is a growing concern in aging aircraft. The purpose of this portion of the study is to detect small loss in thickness using this x-ray technique. This information will help determine the sensitivity attainable using through transmission as a preliminary evaluation for the on going future development of a back-scatter energy-dispersive technique. The back-scatter technique can be used as an in-service technique for the detection of metal corrosion in aging aircraft.

The theory of this energy-dispersive technique is presented. The equations described will be used for the calculations of the results. Since this technique is energy dependent, an optimization study is conducted to help select the two energies of choice for each of the composite systems studied. The error estimation is included as part of the optimization study because it predicts the uncertainties in our experimental measurements. The instrument verification and experimental procedures are then described. X-ray beam collimation for the detector and generator will be presented, and their significance will be discussed. This will be followed by linear attenuation measurement procedures for solids and powders. A novel technique was developed in this study to measure the linear attenuation coefficients of powders. Then we will present sample descriptions of the three composite systems we have mentioned earlier. This is followed by the results and discussions of the experimental measurements. Experimental material characterization to measure thicknesses and densities of the components in the composites are presented. Predicted and experimental results are then compared. Finally, recommendations will be made for future direction of research.

2. THEORY

2.1 Introduction

The physics upon which this technique is based depends on the fact that the x-ray attenuation is a function of both the photon energy and the elemental composition of the material. When a well-collimated narrow beam of photons passes through a homogeneous sample of thickness L , the ratio of the transmitted beam intensity I , emerging from the target along the incident beam direction, to the incident beam intensity I_0 is given by Lambert's law,

$$\frac{I_0}{I} = \exp(\mu L) . \quad (1)$$

This equation assumes that no scattered photons reach the detector, and μ is the linear attenuation coefficient of the element (μ has unit of cm^{-1}). The linear attenuation coefficient is related to the mean-free-path τ by the expression

$$\mu = \tau^{-1} . \quad (2)$$

The mean free path of a photon, of a given energy traveling in an element, is the average distance that the photon can travel without interacting with the element. The mass attenuation is an alternate expression for the attenuation coefficient of an element. The mass attenuation coefficient $\tilde{\mu}$ ($\tilde{\mu}$ has unit of cm^2/gm), involving the density ρ , is given by the expression,

$$\tilde{\mu} = \frac{\mu}{\rho} . \quad (3)$$

When the material consists of a chemical compound or of a homogeneous mechanical mixture such as a composite, the linear attenuation coefficient is given by the weighted sum of the linear coefficients of the components. This is known as the rule-of-mixture. Thus,

$$\mu = \sum_i^k \mu_i w_i , \quad \sum_{j=1}^k w_j = 1 , \quad (4)$$

where μ_i is the linear attenuation of the i -th element and w_i is its proportion by weight. For a chemical compound with the chemical formula $(Z_1)_{a_1}(Z_2)_{a_2}...(Z_n)_{a_n}$, where Z_i 's are the atomic number of the elements, the weighting factor for the i -th element, Z_i , is given by

$$w_i = \frac{a_i A_i}{\sum_{j=1}^n a_j A_j} . \quad (5)$$

where A_i 's are the atomic weight of the element, and a_i 's are the number of atoms of the element in the chemical formula [10].

By using Lambert's law and the rule-of-mixtures, we can obtain sufficient information to determine the constituent materials of the composite by measuring the attenuation of the initial x-ray beam at a number of selected energies as the beam passes through the composite. Therefore, the x-ray attenuation in materials can be expressed in the following general form using Lambert's law and the rule-of-mixtures,

$$\frac{I(E_j)}{I_o(E_j)} = \exp\left(-\sum_i (\mu_i(E_j) X_i)\right) , \quad (6)$$

where i denotes the components in the composite, and j denotes the energies at which the parameters are measured. $I_o(E_j)$ is the incident beam intensity, $I(E_j)$ is the transmitted beam intensity, X_i is the thickness of component i , and $\mu_i(E_j)$ is the linear attenuation coefficient of component i measured at E_j .

We need a minimum of two energies to study a two component composite. This gives us the following two equations,

$$\xi(E_1) = \mu_1(E_1)X_1 + \mu_2(E_1)X_2 \quad (7a)$$

$$\xi(E_2) = \mu_1(E_2)X_1 + \mu_2(E_2)X_2 , \quad (7b)$$

where

$$\xi(E_j) = \ln\left(\frac{I_o(E_j)}{I(E_j)}\right) . \quad (8)$$

The linear attenuation coefficients for elements, or compounds, can be either obtained from experimental measurements or can be obtained from XCOM predictions [3]. Then, by taking the attenuation measurements of the initial beam at selected energies, we have sufficient information to solve the two simultaneous equations (7a,7b) and the results are,

$$X_1 = \frac{\xi(E_1) \mu_2(E_2) - \xi(E_2) \mu_2(E_1)}{\mu_1(E_1) \mu_2(E_2) - \mu_1(E_2) \mu_2(E_1)} \quad (9a)$$

$$X_2 = \frac{\xi(E_2) \mu_1(E_1) - \xi(E_1) \mu_1(E_2)}{\mu_1(E_1) \mu_2(E_2) - \mu_1(E_2) \mu_2(E_1)} \quad (9b)$$

Though the work presented here studies two component composites, this does not exclude the capability of this x-ray technique for studying composites with more than two components. To study such composites, one needs to measure the attenuation of the beam at more energies along the x-ray spectrum according to the number of components.

In making these measurements one has the choice of energies to use. There are many constraints such as x-ray penetration through material, measurement time and maximum energy of the x-ray source that may influence the energy selections. Thus within these constraints, one wishes to optimize the accuracy of the thickness determination. In next section we will derive the expressions for quantitatively assessing the accuracy of the technique outlined above for a two component composite.

2.2 Optimization of energy choices

The energy dependence of this technique makes it necessary to define an optimum energy pair for a two-component composite. An optimum energy pair in Eq.9 will give the minimum uncertainty for the calculated thicknesses, X_i 's. We can derive an expression from the error-propagation analysis theorem to predict the accuracy of the thickness measurements.

Since this dual energy technique is based on calculating the material thicknesses using the linear attenuation coefficients of the materials, any source of error in the linear attenuation coefficients will appear ultimately as error in the thickness determinations. Therefore, we will first derive an expression to evaluate the uncertainty in the experimentally measured linear attenuation coefficients. The expression for calculating the linear attenuation coefficients is given as,

$$\mu = \frac{\ln\left(\frac{I_o}{I}\right)}{X} . \quad (10)$$

In general, in error-propagation analysis we do not know the actual errors in any of the parameters used in the calculations. What we may know instead is some characteristic of the uncertainty or estimated error of each parameter, such as the standard deviation for each parameter. The statistical derivation of the error-propagation analysis is found elsewhere [4]. The uncertainty of the measured linear attenuation coefficient, σ_μ , can be expressed as the sum of the uncertainties of photon intensity measurements and the caliper thickness measurement of the absorbing material, as follows,

$$\frac{\sigma_\mu^2}{\mu^2} = \sigma_x^2 \left(\frac{1}{X}\right)^2 + \frac{\sigma_{I_o}^2 \left(\frac{1}{I_o}\right)^2}{\xi^2} + \frac{\sigma_I^2 \left(\frac{1}{I}\right)^2}{\xi^2} , \quad (11)$$

where

$$\xi = \ln\left(\frac{I_o}{I}\right). \quad (12)$$

We can also express the uncertainty of the calculated thicknesses from Eq. 9 as a sum of the uncertainties of each of the parameters. Equations 9a and 9b are each a function of six independent parameters. Hence, we can express the approximation for the standard deviation for X_1 and X_2 as,

$$\begin{aligned} \sigma_{X_1}^2 = & \sigma_{\mu_1(E_1)}^2 \left(\frac{\partial X_1}{\partial \mu_1(E_1)} \right)^2 + \sigma_{\mu_1(E_2)}^2 \left(\frac{\partial X_1}{\partial \mu_1(E_2)} \right)^2 + \sigma_{\mu_2(E_1)}^2 \left(\frac{\partial X_1}{\partial \mu_2(E_1)} \right)^2 \\ & + \sigma_{\mu_2(E_2)}^2 \left(\frac{\partial X_1}{\partial \mu_2(E_2)} \right)^2 + \sigma_{\xi(E_1)}^2 \left(\frac{\partial X_1}{\partial \xi(E_1)} \right)^2 + \sigma_{\xi(E_2)}^2 \left(\frac{\partial X_1}{\partial \xi(E_2)} \right)^2 \end{aligned} \quad (13a)$$

$$\begin{aligned} \sigma_{X_2}^2 = & \sigma_{\mu_1(E_1)}^2 \left(\frac{\partial X_2}{\partial \mu_1(E_1)} \right)^2 + \sigma_{\mu_1(E_2)}^2 \left(\frac{\partial X_2}{\partial \mu_1(E_2)} \right)^2 + \sigma_{\mu_2(E_1)}^2 \left(\frac{\partial X_2}{\partial \mu_2(E_1)} \right)^2 \\ & + \sigma_{\mu_2(E_2)}^2 \left(\frac{\partial X_2}{\partial \mu_2(E_2)} \right)^2 + \sigma_{\xi(E_1)}^2 \left(\frac{\partial X_2}{\partial \xi(E_1)} \right)^2 + \sigma_{\xi(E_2)}^2 \left(\frac{\partial X_2}{\partial \xi(E_2)} \right)^2. \end{aligned} \quad (13b)$$

Substituting the partial derivatives into Eq.13 and dividing by Eq. 9, we arrive at the following pairs of equations for the fractional thickness uncertainties:

$$\begin{aligned}
\frac{\sigma_{X_1}^2}{X_1^2} = & \left(\frac{\mu_1(E_1) \mu_2(E_2)}{\Delta} \right)^2 \left(\frac{\sigma_{\mu_1(E_1)}}{\mu_1(E_1)} \right)^2 + \left(\frac{\mu_1(E_2) \mu_2(E_1)}{\Delta} \right)^2 \left(\frac{\sigma_{\mu_1(E_2)}}{\mu_1(E_2)} \right)^2 \\
& + \left(\frac{\mu_2(E_1) \mu_1(E_2)}{\Delta} - \frac{\mu_2(E_1) \xi(E_2)}{\mu_2(E_2) \xi(E_1) - \mu_2(E_1) \xi(E_2)} \right)^2 \left(\frac{\sigma_{\mu_2(E_1)}}{\mu_2(E_1)} \right)^2 \\
& + \left(\frac{\mu_1(E_1) \mu_2(E_2)}{\Delta} - \frac{\mu_2(E_2) \xi(E_1)}{\mu_2(E_2) \xi(E_1) - \mu_2(E_1) \xi(E_2)} \right)^2 \left(\frac{\sigma_{\mu_2(E_2)}}{\mu_2(E_2)} \right)^2 \\
& + \left(\frac{1}{1 - \frac{\mu_2(E_1) \xi(E_2)}{\mu_1(E_2) \xi(E_1)}} \right)^2 \left(\frac{\sigma_{\xi(E_1)}}{\xi(E_1)} \right)^2 + \left(\frac{1}{1 - \frac{\mu_2(E_2) \xi(E_1)}{\mu_2(E_1) \xi(E_2)}} \right)^2 \left(\frac{\sigma_{\xi(E_2)}}{\xi(E_2)} \right)^2
\end{aligned} \tag{14a}$$

and,

$$\begin{aligned}
\frac{\sigma_{X_2}^2}{X_2^2} = & \left(\frac{\mu_2(E_2) \mu_1(E_1)}{\Delta} - \frac{\mu_1(E_1) \xi(E_2)}{\mu_1(E_1) \xi(E_2) - \mu_1(E_2) \xi(E_1)} \right)^2 \left(\frac{\sigma_{\mu_1(E_1)}}{\mu_1(E_1)} \right)^2 \\
& + \left(\frac{\mu_2(E_1) \mu_1(E_2)}{\Delta} - \frac{\mu_1(E_2) \xi(E_1)}{\mu_1(E_1) \xi(E_2) - \mu_1(E_2) \xi(E_1)} \right)^2 \left(\frac{\sigma_{\mu_1(E_2)}}{\mu_1(E_2)} \right)^2 \\
& + \left(\frac{\mu_1(E_2) \mu_2(E_1)}{\Delta} \right)^2 \left(\frac{\sigma_{\mu_2(E_1)}}{\mu_2(E_1)} \right)^2 + \left(\frac{\mu_1(E_1) \mu_2(E_2)}{\Delta} \right)^2 \left(\frac{\sigma_{\mu_2(E_2)}}{\mu_2(E_2)} \right)^2 \\
& + \left(\frac{1}{1 - \frac{\mu_1(E_1) \xi(E_2)}{\mu_1(E_2) \xi(E_1)}} \right)^2 \left(\frac{\sigma_{\xi(E_1)}}{\xi(E_1)} \right)^2 + \left(\frac{1}{1 - \frac{\mu_1(E_2) \xi(E_1)}{\mu_1(E_1) \xi(E_2)}} \right)^2 \left(\frac{\sigma_{\xi(E_2)}}{\xi(E_2)} \right)^2,
\end{aligned} \tag{14b}$$

where

$$\Delta = \mu_1(E_1) \mu_2(E_2) - \mu_1(E_2) \mu_2(E_1) .$$

We have shown in our previous study [27] that the uncertainties due to beam fluctuations can be neglected when more than a million photons are counted. In our experiment we integrated the incident and transmitted x-ray beam intensities over time to achieve high counting statistics of over a million counts, and hence we ignored the uncertainties contributed by the beam intensities in our analysis. The dominating fundamental source of error comes from the uncertainties in our experimentally measured linear attenuation coefficients. In turn,

these linear attenuation coefficient uncertainties are dominated by the caliper thickness measurements of the absorber. Thus, out of the six original influencing parameters in Eq. 14, we are left with four significant parameters in our error propagation analysis. These parameters are the linear attenuation coefficients of the two materials at the two choices of energies.

The optimum energy-pair, predicted by the error-propagation analysis for the minimum uncertainty, can be found when the two energies are farthest apart from one another, even for cases where the linear attenuation curves intersect one another. The low energy should be taken, if possible, where the difference in the linear attenuation coefficients is the greatest between the two materials. This gives maximum contrast for discriminating between the two materials. The high energy measurement should be made where the difference in the linear attenuation coefficient between the two materials is the least. Measuring at the high energies effectively determines the total thickness of the composite, because the attenuation coefficients of most materials are similar at these energies. Additional constraints in the choice of energies are experimental constraints, such as the time required to accumulate high counting statistics and the ability to accurately measure the attenuation coefficients at the two energies of choice.

Care must be taken in selecting the low energy, E_1 , and high energy, E_2 . The upper X-ray energy of the optimum energy pair is naturally constrained by the power output of the X-ray generator. In addition, measurements made in the upper x-ray energy range are constrained by the contrast between the transmitted and incident beam intensities, which can be explained through Eq. 11. At high energies the linear attenuation coefficients are low for all elements, therefore the difference between the incident and transmitted beam intensities becomes small. This means the value of ξ is small, and the uncertainty contribution due to I_0 and I can be as large as the uncertainty in the caliper thickness measurement; all this gives rise to large uncertainties in the linear attenuation coefficient measurements. In contrast, at low energies, the linear attenuation coefficients are large, consequently, the uncertainties due to the transmission

ratio are small. At low energies the uncertainty in the linear attenuation coefficient is sensitive to the caliper thickness measurement, X . A large absorber thickness will minimize the uncertainty due to caliper thickness measurements, but it may introduce low transmission beam intensities. Thus, an absorber thickness should be chosen to closely approach the optimum transmission ratio, (I_0/I) , of 3 at the low energies [24].

3. INSTRUMENT CHARACTERIZATION

3.1 High-purity germanium detector

All the measurements made in this study have been performed using a High-Purity germanium detector (HP-germanium detector) coupled to a multichannel pulse-height analyzer (MCA). The HP-germanium detector is a semiconductor x-ray detector. The detector converts x-ray photons into electrical pulses; the amplitude of the electrical pulse generated in the HP-germanium detector is proportional to the x-ray energy deposited by the photons. The electrical pulses from the germanium crystal are fed into a preamplifier and then to an amplifier that boosts the amplitude of the pulse to a positive volt signal ranging from 0 to 10 volts. The shaping time of the amplifier is set to 1 μ sec to enable high counting rate. The output signal from the amplifier is suitable for use with a multichannel analyzer. The MCA is initialized to accept 5 keV to 160 keV with 2048 channels. This enables the MCA to monitor the pulse counts for each channel number for a preset count time.

HP-germanium detector is unlike many other detectors because it is cooled to liquid nitrogen temperature of 77 K. The inconvenience in maintaining this constant cryogenic temperature is outweighed by the benefit of the energy resolution obtained from the germanium detector. This sensitivity is not obtainable using x-ray detectors such as NaI or CsI. The HP-germanium detector has an energy discriminating resolution of 0.57 keV FWHM (Full Width at Half Maximum) at 60 keV, as shown in Fig. 3.1, whereas the energy resolution of a common x-ray detector such as NaI is about 5.4 keV FWHM for the same energy (see Fig. 3.2).

Figure 3.1. Germanium detector resolution of Americium 241 isotope at 60 keV

Figure 3.2. Sodium iodide detector resolution of Americium 241 isotope at 60 keV

3.2 Collimators

Collimators in the experimental setup are used to define the narrow pencil beam configuration that is necessarily assumed from Lambert's law. The collimation setup also suppresses the background due to scattered x-rays from non-essential objects in the x-ray vault. Thus the collimation defines the volume of the object probed by the x-ray beam.

The HP-germanium detector is shielded in a quarter inch thick lead box as shown in Fig. 3.3. A collimator hole of 350 micron diameter defines the diameter of the probed volume on the sample. This also reduces the intensity of incident, and scattered, photons reaching the detector. The detector collimation reduces the intake of scattered photons to be within a cone-arc of 1.06 degrees, approximately.

It is important to shield the non-essential objects, that would typically be illuminated by the x-ray generator, scattering incident x-rays that may reach the detector. Non-essential objects lying between the source and the detector are shielded from the beam by the source-collimation as indicated in Fig 3.4. The source-collimator essentially eliminates x-ray scattering from non-essential objects that would typically be illuminated by the x-ray source by reducing the cone-beam from 30 degrees to 1.9 degrees. It is also important to note the presence of the detector-collimator and the positioning of the object between the source and the detector; because, in some cases the coherent and incoherent scattering may reach the detector, giving too high a value for the transmitted intensity. The detector-collimator determines the volume of the object probed. Even so, the detector also picks up signals of scattered x-rays from the scattering-volume in the object. One can reduce much of this scattering volume by positioning the object closer to the detector. A smaller collimation hole can equally reduce the scattering volume on the object, but this would also proportionally reduce the incident X-ray beam intensity. If a radioactive isotope source were used it would be necessary to balance the collimator size for the detector while maintaining sufficiently high incident x-ray beam intensity for statistical

Figure 3.3. Detector collimation and its influence on the probed and scattered volume on the object

Figure 3.4. X-ray source collimation and its effect on scattered radiation

purposes. By using an x-ray generator as the source, we can overcome this statistical limitation by increasing the current generating the x-ray beam.

3.3 Positioner and controller program

The block diagram of the experimental setup of the electronic communication between the PC and the detector and positioner is shown in Fig. 3.5. The positioners (Daedal Inc.) are used to move the sample object during an experiment by precision stepper motors (Compumotor Inc.). The motors are controlled with a DELL 386 PC using a Compumotor PC-23 indexer board placed in the computer. The indexer board sends commands to, and receives positioner status from, the motor adapter. The adapter in turns sends commands to the motor, through a motor servo, instructing how much the motor should rotate for a given travel distance. The stepper motor has stepping resolution of 100,000 steps per inch. This gives us a precision of 10 μ inch to move the sample back and forth on the positioner.

A typical experimental measurement in this study takes up to 24 hours to acquire sufficient statistics of over a million counts for the beam intensities, I and I_0 . By moving the object in and out of the x-ray beam, the program can monitor the x-ray beam instability over time. The two beam intensities are integrated over time to obtain the transmission ratios. Therefore a controller program was developed to automatically move the sample on the positioner in and out of the x-ray beam at preset time intervals to collect the transmitted and the incident beam intensities, respectively. The controller program was developed to allow the operator to interactively create an energy bin array with tailored energy bin sizes. The positioner movement and data acquisition from the MCA are controlled using the PC with a C++ driver program called "MCA_2."

Figure 3.5. Germanium digital detector instrumental setup

3.4 X-Ray source

The x-ray source used in this work is a Ridge HOMX 160A microfocus generator with a 10 micron focal spot size approximately. The generator provides a Bremsstrahlung spectrum to measure the attenuation coefficients. The energies of the spectrum are resolved using the MCA. The generator is capable of operating at energies up to 160 keV and with beam currents of up to 2 mA. It should be mentioned that this energy sensitive technique was also successfully demonstrated with the use of radioisotope sources [27]. Therefore the microfocus capability is not a required feature for the x-ray generator; indeed, any radiation sources could have been used for this study.

3.5 HP-Germanium digital detector calibration

Calibration of the HP-germanium digital detector with the MCA setup is necessary to correlate the MCA channels to the x-ray energies. We used radioactive isotopes to calibrate the detector system. Radioactive isotopes emit well-defined x-ray energies and these energies show up as sharp peaks on the MCA spectrum. Thus, a linear detector calibration curve can be developed with sufficient number of radioactive isotopes as plotted in Fig 3.6. The present energy sensitivity of the MCA in this study is 12 channels per keV.

3.6 Amplifier pile-up rejecter

X-ray spectral distortion is often found during high counting rates. This spectral distortion caused by signal pile-up usually occurs when two or more x-ray photons strike the detector at close time intervals. In such instances, the germanium detector electronics is unable to resolve each signal generated by the photons. Thus the electrical signals are summed into one larger signal. This count rate limitation is strictly related to the number of input counts per unit of time and is independent of energy [1]. Consequently, the spectral distortion in the

Figure 3.6. HP-Germanium digital detector calibration curve

Bremsstrahlung spectrum will show more photon counts at higher energies than in reality. This is a problem because this x-ray energy-dispersive technique is energy sensitive. The Canberra amplifier used here has an efficient pile-up rejecter (PUR). The PUR provides an output logic pulse for the associated multichannel analyzer to suppress the spectral distortion caused by high counting rates. The effect of the PUR on the spectrum can be seen in Fig 3.7.

Figure 3.7. The influence of pile-up-reject on MCA spectrum

4. SAMPLE DESCRIPTION

4.1 Introduction

Three different composite systems were investigated in this study. The first composite system is a graphite-epoxy composite, consisting of two materials, graphite-fibers and epoxy-resin, having very similar elemental compositions and densities. The second composite system is a bone-tissue composite, consisting of two materials having very different elemental compositions and densities. The third is a composite system of aluminum 2024 metal and its corrosion product composed of two materials with similar elemental composition, but very different densities. The following subsections describe the materials used in the composites and the quantitative composition of each of the components used in the composite samples.

4.2 Graphite-epoxy composite

Graphite-fiber-reinforced-epoxy composite (graphite-epoxy composite) is increasingly replacing metal alloys because the selective directional strength of the composite can be tailored to match the required strength performances for load bearing; while, at the same time, the composite material is lighter than metal alloys having comparable strength.

The graphite-epoxy composite used in this study is made from Magnamite 8551-7 uniaxial prepreg sheets manufactured by Hercules Inc. This composite is considered to be the next-generation composite because this amine-cured epoxy-resin is doped with micro-spheres. This makes the composite more impact resilient. The graphite fiber in Magnamite 8551-7 is a synthetic polyacrylonitrile (PAN-based) graphite fiber known as IM-7 fiber. Samples of neat-epoxy-resin were provided by Hercules Inc. for linear attenuation coefficient measurements. We were also able to obtain some graphite fibers, but the graphite fibers have diameter of 5

μm , and this made the linear attenuation coefficient measurement difficult. The determination of the linear attenuation coefficients for the materials is deferred to Section 5.

A sample of Magnamite composite was fabricated at Iowa State University's Aerospace Engineering and Engineering Mechanics departmental composite laboratory. The lay-up and curing procedure for the Magnamite composite follows that described in the International Encyclopedia of Composites [13]. The composite lay-up is 0/90 with a total of 23 layers. The final thickness of the composite is 0.457 cm after curing.

A coupon from the Magnamite composite, size 2.5 cm by 2.5 cm, by 0.457 cm thick, is analyzed to determine the effective total thickness of the IM-7 fibers and epoxy-resin in the 23 layers. This was needed for comparison with the x-ray measured thickness values. A corner of the composite coupon was chosen for analysis. The assumptions are that the fiber-to-resin ratio variation at the localized corner of the coupon is small, and that the fibers are perpendicular to the side-faces of the corner.

The two adjacent surfaces to the corner were polished to produce surfaces that exposed the ends of the fibers. We took black-and-white photomicrographs at layers that have exposed fiber-ends. The photomicrographs are taken at a magnification of 300. Two arrays, of 6 columns by 12 rows, and 6 columns by 13 rows, of photomicrographs are taken of the two polished surfaces on either side of the selected corner. The two arrays covered an area of 0.310 cm by 0.457 cm, approximately, on either side of the corner.

An image processing technique is used to determine the fiber-to-resin ratio in each photomicrograph. The black-and-white photographs are digitized using a camera-digitizer; the image is acquired through a PC based image acquisition software. The digitized data are transferred to an image analysis software based on a Macintosh computer. A black-and-white thresholding technique is used to determine the fiber-to-resin ratio in each photomicrograph. The fiber-to-resin ratios of the micrographs are averaged in 12 columns in the thickness

direction. The variation in the fiber-to-resin ratios is 1.4% across the 12 columns. This verifies that the fiber-to-resin variation is indeed small in a localized region for this composite coupon. The averaged percentage of fiber and resin, by area, for the 12 columns is 55.5% and 44.5% respectively. Thus the effective total thicknesses for graphite IM-7 fibers and epoxy-resin are 0.254 cm and 0.203 cm respectively.

The fiber-to-resin ratios in the composite samples are varied by adding known thicknesses of neat-epoxy-resin to the composite coupon. The compositions of the samples are shown in Table 4.1.

Table 4.1. Composition of graphite-epoxy composite samples

Composite #	Fiber-to-resin ratio	Total thickness of epoxy-resin , cm	Total thickness of fiber, cm
1	1.249	0.203 ± 0.008	0.254 ± 0.008
2	1.025	0.248	0.254
3	0.989	0.257	0.254
4	0.908	0.271	0.254
5	0.803	0.316	0.254
6	0.704	0.361	0.254
7	0.515	0.493	0.254

4.3 Bone-Plexiglas composite

In this part of the work, we applied this x-ray energy-dispersive technique to evaluate its sensitivity for detecting the onset of osteoporosis. Osteoporosis is the most common of all bone diseases in adults, especially in old age. Usually in osteoporosis the osteoblast cell activity is less than normal, and consequently the rate of bone deposition is depressed. Thus in osteoporosis the rate of bone loss is greater than the rate at which bone is deposited, this gives an over all net loss of bone material. Osteoporotic process is typically slow, occurring over periods of months or years, thus the variation due to bone loss can be very small. Early

detection of bone loss can give information to avoid physical stress that can result in bone fractures.

If we classify the body parts of a human in terms of their respective densities, we can, in general, view the human body as a two component composite consisting of bone and soft-tissues (flesh). In this study we wish to determine the thicknesses of flesh and density of bone in our samples using this x-ray energy-dispersive technique for detecting the onset of osteoporosis.

We made our own samples to simulate body parts of a human subject. We used dense bone samples from a cow to simulate human bone in our study. A cattle femur bone was cut to produce parallel surfaces where the thicknesses are known. The density of the cattle bone sample is 2.077 gm/cm^3 . This is determined from mass and volume measurements of the bone sample. Plexiglas is used to simulate soft-tissue in this study. Plexiglas is chosen because it will not decompose like meat at room temperature during our long data acquisition time. In addition, Plexiglas is commonly used as a standard calibration material, for the same reason mentioned above, in medical X-ray CAT scan to simulate soft-tissue.

Two sets of samples are made to simulate two different parts of a human patient. One set of samples has the bone and Plexiglas thicknesses of 0.808 cm and 0.925 cm respectively. This set of samples simulates the fingers of the patient. Another set of samples simulates the calf of the patient. The thicknesses of the second set of samples of bone and Plexiglas are 2.400 cm and 1.834 cm respectively. Two sets of samples were investigated to determine the accuracy of this x-ray technique to detect the onset of osteoporosis on different body parts with different thicknesses. During osteoporosis, the outer diameter of the bone does not change; only the density of the bone changes. Similarly, then, we can have the outer dimensions of the bone (0.808 cm and 2.400 cm) in the two sets of bone samples remain the same. Then, by

Table 4.2. Composition of bone-Plexiglas composite samples

Sample #	% Osteoporosis (% bone loss)	Bone density, gm/cm ³	Thickness of bone, cm	Thickness of Plexiglas, cm
a) Sample set 1				
1	0.0	2.077	0.808 ± 0.001	0.925 ± 0.001
2	20.4	1.653	0.643	0.925
3	27.9	1.496	0.582	0.925
4	34.6	1.358	0.528	0.925
b) Sample set 2				
1	0.0	2.077	2.400 ± 0.001	1.834 ± 0.001
2	34.6	1.358	1.569	1.834
3	46.9	1.102	1.274	1.834
4	56.5	0.903	1.044	1.834
5	68.6	0.653	0.754	1.834

removing known thicknesses of bone from the samples, we can effectively change bone density, simulating osteoporosis. The density samples have compositions shown in Table 4.2.

In our study, we assume that we can obtain the total cross-sectional thickness on a body part of a patient by a caliper. There can be a simple caliper-like mechanism that measures the thickness of the body part whilst the same mechanism holds the body part steady during x-ray measurements. Thus the total thickness of the body part, X_{total} , is given as,

$$X_{\text{total}} = X_b + X_f, \quad (15)$$

where X_b and X_f are the thicknesses of bone and flesh respectively.

For the purpose of measuring the bone density, we can express Lambert's equation from Eq. 6 as the following,

$$\xi(E_i) = \mu_f(E_i)X_f + \tilde{\mu}_b(E_i)\tau_b \quad , \quad (16)$$

where $\xi(E_i)$ is the beam attenuation measured at energy E_i , $\mu_f(E_i)$ is the linear attenuation coefficient of flesh, measured at energy E_i , X_f is the thickness of flesh, $\tilde{\mu}_b(E_i)$ is the mass attenuation coefficient of bone measured at energy E_i , and τ_b is the area-weight of bone (area-weight has units of gm/cm^2). We can use Eq. 16 in the same way as the dual energy technique described in Section 2. Instead of obtaining the thicknesses of Plexiglas and bone from the calculation, we determine the thickness of Plexiglas and the area-weight of the bone. We can then subsequently determine the density of the bone, ρ_{bone} , using the following equation,

$$\rho_{\text{bone}} = \frac{\tau_b}{X_{\text{total}} - X_f} \quad . \quad (17)$$

4.4 Aluminum-aluminum corrosion composite

NDE techniques are ideal for detecting corrosion during aircraft maintenance because access to corroded areas is frequently limited or impossible due to intervening structures. Corrosion is a concern in aircraft skin of aging aircraft that are still in use. Aircraft skins are generally made of 2024 aluminum alloy sheet metals that are 40 mils thick. The goal of this portion of the work is to detect the material loss of less than 10% in aircraft aluminum skin using this energy-dispersive technique. NDE inspection techniques are often conducted from one side of the structure - the outer surface of the aircraft. Although, this x-ray energy-dispersive technique requires access to both sides of the aircraft skin structure, the long term goal is to develop a Compton backscatter technique for quantitative determination of corrosion.

Table 4.3. Composition of aluminum-aluminum corrosion composite samples

Sample #	% Material loss in Al 2024	Aluminum 2024 thickness (mils)	Aluminum hydroxide thickness (mils)
1	0.0	40.00±0.50	0.0
2	1.3	39.50	0.50 ± 0.50
3	3.8	38.50	1.50
4	5.5	37.80	2.20
5	6.5	37.40	2.60
6	10.0	36.00	4.00
7	15.0	34.00	6.00
8	23.8	30.50	9.50
9	26.3	29.50	10.50
10	36.3	25.50	14.50
11	38.8	24.50	15.50
12	50.0	20.00	20.20

Therefore, it is desirable to determine the sensitivity attainable using a more conventional transmission set up before implementing a more complicated backscatter technique.

Aluminum 2024 metal and its corrosion product can be viewed as two materials in a composite system. The composition of aluminum 2024 alloy consists of 93.6% Al, 4.4% Cu, 0.5% Mn and 1.5% Mg [15]. The corrosion process of aluminum 2024 is simply the oxidation of the aluminum metal. We assume that the corrosion product is formed under a basic pH condition, thus the corrosion product can be approximated by aluminum hydroxide ($\text{Al}(\text{OH})_3$).

In this study, we made our own aluminum-aluminum corrosion samples having the composition shown in Table 4.3. The corrosion simulation samples are made by machining away a channel 0.5 inch wide and 0.5 inch long of known thickness of aluminum 2024 metal from a total thickness of 40 mils. A microscope cover-glass is glued over the face of the

channel. Then, the space beneath the cover-glass is ultrasonically packed with aluminum hydroxide powder. The thickness of the cover-glass is approximately 8 mils. During beam attenuation measurements on the corrosion samples, another cover-glass with identical thickness is used to attenuate the incident beam. This is done to compensate the attenuation effect of the cover-glass in the beam attenuation measurements.

5. LINEAR ATTENUATION COEFFICIENT MEASUREMENT

5.1 Linear attenuation measurement for solids

In its passage through a material, a beam of photons may as a result of interactions be either absorbed or scattered. There are three types of interaction in the typical range of energies found in x-ray NDE inspection (10-350 keV): photoelectric absorption, elastic scattering (Raleigh scattering) and inelastic scattering (Compton scattering). A combination of such interactions gives the total linear attenuation coefficients as shown in Fig.5.1. Absorption may be thought of as the disappearance of a photon from the beam, and scattering as a combination of absorption and emission. The details of these processes can be found in the literature [9]. The total attenuation coefficient of a material is then the probability that a photon being removed from the beam in the absorber, and they are a function of energy.

One can measure the linear attenuation coefficient at a particular energy by using radioactive isotope sources. By plotting the transmitted beam intensity as a function of thickness, at a particular energy, one can obtain the linear attenuation coefficient by curve fitting an exponential line to the data. Figure 5.2 shows a plot of transmitted beam intensities through aluminum as a function of thickness for three different isotope energies. The exponential slopes are the linear attenuation coefficients for the three energies. This process of measuring attenuation coefficients is tedious. The linear attenuation coefficients can be measured much more easily and quickly using a Bremsstrahlung source and a germanium detector. One can obtain a continuous linear attenuation coefficient curve across the x-ray spectrum with a single measurement for the incident and transmitted beam intensities. The experimental setup for this measurement was described in section 3.1.

Figure 5.1. Energy dependence of various x-ray interaction processes in aluminum

Figure 5.2. Measured aluminum linear attenuation coefficients using radioactive isotopes

We have relied on the values for linear attenuation coefficient measured by us because there are differences in the values of attenuation coefficients in the literature [11]. Typical referenced attenuation values have uncertainties of 5% to 10% [19]. Deslattes has suggested that one should not rely on international tables for attenuation coefficients to better than 10% [6]. In addition, it is important that the transmission attenuation measurements be made with the same experimental setup as for the beam attenuation coefficient measurements. This is because differences in beam attenuation due to beam geometry can greatly effect the energy-dispersive measurements. In particular, one should not use "broad" beam attenuation coefficients for calculation with "narrow" beam transmission measurements, and vice versa.

In accordance with Lambert's law, the experimental setup requires narrow "pencil-beam" geometry, i.e., both the source and detector being well collimated. The positioning of the absorber immediately in front of the detector was chosen because it effectively minimized the scattered radiation, from the absorber, from reaching the detector. Thus the accuracy of the linear attenuation coefficient and beam attenuation measurements, for the absorber will be dependent on how much of the small angle scattering is registered as part of the transmitted beam intensities.

The collimated x-ray beam is recorded with the HP-germanium detector connected to a multichannel pulse-height analyzer. The absorber was placed between the x-ray source and the detector, with the absorber surface perpendicular to the x-ray beam. If a thick absorber is placed in the beam, the transmitted beam intensity will be low and a relatively long time will be required to accumulate sufficient counts. In fact, for undesirable low transmissions, the magnitude of the transmitted beam may be comparable to the background. On the other hand, if a very thin absorber is used, the transmitted beam intensity approaches that of the incident beam intensity, and the transmission ratio becomes unfavorable. In our experiment some intermediate transmission ratio was employed. The absorber thickness was chosen to give a

transmission (I_0/I) value of 1.1 to 5, thus closely approaching the optimum attenuation condition with transmission ratio of 3 [24].

The value of the linear attenuation coefficient was calculated using the caliper measured thickness of the absorber, according to Eq. 10. A correction for air displaced by the absorber thickness is negligible for solid absorber, because the magnitude of the air attenuation is negligible compared to the solid. Rather, the main uncertainty in our linear attenuation measurements is from measuring the thickness of the absorber specimens using a caliper. From equation 12 we find that the uncertainty in the absorber thickness is the dominating term for low energy X-rays, because the transmission ratio is high at these energies. On the other hand, the contribution due to the absorber thickness and beam intensity measurements can be nearly equal for high energy X-rays when the transmission ratio is small at these energies [7].

A series of measurements with the absorber in and out of the beam was taken to obtain the transmitted beam and incident beam intensities respectively. Each set of intensities was recorded for a preset live-time at an averaged count rate of 8 kHz or less. Figure 5.3 indicates the measured attenuation coefficient is slightly count rate dependent. This observation was also made by Gerward in his attenuation measurements [7]. Thus the detector system is operated in the upper-most linear region of the detector count-rate curve, at about 8 kHz as shown in Fig. 5.4. The stability of the X-ray generator is monitored with the scanning program "MCA_2." The results of our measurements did not indicate any fluctuations larger than those that can be accounted for by the usual Poisson statistics. The background correction in the data was not required in our attenuation measurements, because the background radiation is registered in the incident and the transmitted beams. Therefore the background radiation will be subsequently normalized as we take the transmission ratio of the beams.

Figure 5.3. Count rate dependence of attenuation coefficients

Figure 5.4. Input pulse rate effect on MCA output

5.2 Linear attenuation measurements for powders

There is an intrinsic difficulty in determining the attenuation coefficient of powders because it is difficult to determine the thickness that should be used in calculating attenuation coefficients. In this thesis work, we have developed a novel technique to measure the attenuation coefficients for the powders. The powder is first ultrasonically packed into a glass vial. The glass vial was thin and the diameter of the vial was uniform. A thin glass vial attenuates less than a thick glass vial; thus one can maintain the high counting statistics in the transmitted x-ray beam. We calculated the packed density of the powder by measuring the mass of the powder in the vial and the volume of the vial. Then by taking an identical glass vial of the same dimensions, we measured the incident and transmitted x-ray beams through the empty and packed vials, respectively, to obtain the transmission ratio.

The major uncertainties for the linear attenuation coefficient measurements for the powders come from the caliper measurements of the inner diameter of the vial and the packed-density of the powder in the vial. The inner diameter of the vial is used to calculate the linear attenuation coefficient of the packed powder. Thus we encounter the similar caliper thickness uncertainty as we did for measuring the attenuation coefficients for solids. In addition to this, we also have uncertainty due to the packed density measurements. It is also possible that the packed density of the powder in the vial is non-uniform. Therefore care must be taken in making the dimensional measurements for the linear attenuation calculations.

5.3 Experimental linear attenuation coefficient measurements

5.3.1 Graphite-epoxy composite

There is no published linear attenuation data for the composite IM-7 fibers. This is because the individual fibers are only 5 microns in diameter. Therefore, we are not able to measure the linear attenuation coefficients for the IM-7 fibers directly with the procedure

described in section 5.1. It is difficult to measure the total thickness of a bundle of fibers to calculate the linear attenuation coefficients. Although we can generate the mass attenuation coefficients for the IM-7 fiber based upon its chemical composition using XCOM, we do not know the density of the IM-7 fibers; therefore we can not convert the mass attenuation coefficients to the linear attenuation coefficients. Therefore, an indirect technique has to be used to determine the linear attenuation coefficients for the IM-7 fibers. The indirect method uses the linear attenuation coefficients of epoxy-resin and the beam attenuation of the characterized Magnamite composite coupon to extrapolate the linear attenuation coefficients of the IM-7 fibers, using Eq. 7. The extrapolated linear attenuation coefficients for the IM-7 fibers and the measured linear attenuation coefficients for epoxy-resin are tabulated in Table 5.1, and they are plotted in Fig. 5.5.

We were able to obtain three woven composite samples of IM-7, AS4 and Carbolon, graphite fibers. IM-7 and AS4 are PAN-based graphite fibers, which differ with Carbolon, which is a pitch-based graphite fiber, as defined based on manufacturing technique. The fibers are essentially carbon based fibers with slightly different elemental compositions. The pitch-based graphite fibers are derived from petroleum carbon polymer precursors, while the PAN-based fibers are made from synthetic carbon polymer precursors. The fibers can differ in fiber density, even among the PAN-based fibers. Although we are still unable to determine the effective total thickness of the fiber in the bundles of woven fiber samples, we did measure the effective-attenuation coefficients for the three graphite samples. This is a measure of the transmission ratio divided by the thickness of the fiber bundle. The thickness includes the air space between the woven fibers.

We determined the degree of confidence in our extrapolated linear attenuation coefficient for IM-7 graphite fibers by dividing it by the effective-attenuation coefficients of each of the three fiber samples. The slopes of the ratios, as a function of energy, would be

Figure 5.5. Experimentally measured linear attenuation coefficients for epoxy-resin and IM-7 graphite fiber

small if the chemical composition of the two materials is similar. This is because x-ray attenuation is a function of the elemental composition of the compound, whereas, if the composition of the materials is different, the slope of the ratio will be large. In Figure 5.6, we see that the slope of the ratio obtained with IM-7 is the smallest. The slope obtained from AS4 is four times the ratio obtained with IM-7. Meanwhile, the slope of the ratio obtained with Carbolon effective-attenuation is larger than the two previous slopes. This difference is expected because the three fibers are made with two different manufacturing techniques. With this analysis we have shown the confidence in our extracted IM-7 graphite fiber linear

Table 5.1. Experimentally measured linear attenuation values of epoxy-resin and graphite IM-7 fibers

Energy (keV)	μ of epoxy-resin (cm-1) ^a	μ uncertainty for epoxy-resin (%) [*]	μ of IM-7 fibers (cm-1) ^a	μ uncertainty for IM-7 fibers (%) [*]
13.5	2.72	4.9	1.64	6.8
15.5	2.01	1.8	1.23	6.8
17.5	1.49	7.1	0.960	2.0
19.5	1.155	0.6	0.773	0.6
21.5	0.915	0.2	0.659	0.4
23.5	0.753	0.2	0.580	0.2
25.5	0.639	0.2	0.516	1.9
27.5	0.555	0.3	0.478	0.8
29.5	0.491	0.3	0.442	0.0
31.5	0.449	0.5	0.412	1.8
33.5	0.411	1.0	0.400	0.1
35.5	0.378	0.8	0.385	0.8
37.5	0.359	0.3	0.368	2.2
39.5	0.342	0.4	0.354	0.6
41.5	0.318	2.2	0.354	2.8
43.5	0.301	2.0	0.344	4.4
45.5	0.297	2.2	0.334	1.1
47.5	0.291	1.2	0.325	1.0
49.5	0.275	2.2	0.325	3.7
51.5	0.275	0.8	0.316	1.3
53.5	0.262	1.3	0.320	4.0
55.5	0.263	0.9	0.307	2.0

a. number of experimental measurements $n = 3$.

* one standard deviation

Figure 5.6. Ratios of extrapolated linear attenuation coefficient of IM-7 fiber divided by the effective attenuation coefficients of 3 different kinds of fibers: IM-7, AS-4 and Carbolon fibers

attenuation coefficients.

We generated the mass attenuation coefficients for a PAN-based graphite fiber from XCOM using the chemical compositions given by International Encyclopedia of Composites [13]. We discovered that the mass attenuation coefficient slope, from XCOM, did not agree with the slope in the extrapolated attenuation coefficient slope. The slope of the ratio obtained from dividing the extrapolated coefficients by XCOM coefficients are too large to show agreement between the two attenuation slopes (see Fig. 5.7). This further demonstrated that it is necessary to measure one's own linear attenuation coefficients for a material.

Table 5.2. Experimentally measured linear attenuation coefficients for bone and Plexiglas

Energy (keV)	μ of bone (cm^{-1}) a	μ of Plexiglas (cm^{-1}) b
31.5	2.670	0.333
33.5	2.277	0.314
35.5	1.967	0.296
37.5	1.726	0.284
39.5	1.531	0.275
41.5	1.367	0.264
43.5	1.229	0.258
45.5	1.116	0.252
47.5	1.022	0.246
49.5	0.937	0.242
51.5	0.874	0.237
53.5	0.807	0.232
55.5	0.757	0.231
57.5	0.709	0.226
59.5	0.672	0.223
61.5	0.651	0.222
63.5	0.614	0.218
67.5	0.564	0.215
69.5	0.548	0.213

a. The uncertainty due to thickness measurement is 1.0%.

b. The uncertainty due to thickness measurement is 0.3%.

Figure 5.7. Ratio of extrapolated linear attenuation coefficient of IM-7 fiber divided by the mass attenuation coefficient of PAN-based graphite generated from XCOM

5.3.2 Bone-Plexiglas composite

We directly measured the linear attenuation coefficients for bone and Plexiglas using procedures described in section 5.1. The thicknesses of bone and Plexiglas were 0.808 cm and 0.925 cm respectively. The linear attenuation coefficients for the bone and Plexiglas are given in Table 5.2 and they are also plotted in Fig. 5.8.

5.3.3 Aluminum-aluminum corrosion composite

We directly measured the linear attenuation coefficients for aluminum 2024 and aluminum hydroxide using procedures described in section 5.1 and 5.2, respectively. The thicknesses of aluminum 2024 and aluminum hydroxide packed vial are 0.102 cm and 1.100 cm respectively. The linear attenuation coefficients for aluminum 2024 and aluminum hydroxide are given in Table 5.3 and they are plotted in Fig. 5.9.

Table 5.3. Experimentally measured linear attenuation coefficients for Al 2024 and Al(OH)₃

Energy (keV)	μ of Al 2024 (cm ⁻¹) a	μ Uncertainty for Al 2024 (%) *	μ of Al(OH) ₃ (cm ⁻¹) a	μ Uncertainty for Al(OH) ₃ (%) *
15.5	23.5	2.0	1.71	17.8
20.5	11.9	0.8	1.91	1.3
25.5	6.65	0.8	1.19	1.5
30.5	4.09	0.8	0.81	1.5
35.5	2.73	1.6	0.59	1.5
40.5	1.96	1.7	0.48	1.5
45.5	1.49	2.2	0.40	1.7
50.5	1.18	0.7	0.36	1.6
55.5	0.97	2.4	0.31	4.1
60.5	0.84	3.3	0.29	5.2

a. number of experimental measurements $n = 3$.

*, one standard deviation.

Figure 5.8. Measured linear attenuation coefficients for bone and Plexiglas

Figure 5.9. Experimentally measured linear attenuation coefficients of aluminum 2024 and aluminum hydroxide are compared with XCOM values

6. RESULTS AND DISCUSSIONS

6.1 Introduction

As mentioned in the Section 2, we need to optimize the choice of energy-pair used in this energy-dispersive technique. Since there are three composite systems in this thesis, we need three different optimum energy-pairs for the three studies. The derived error-propagation expressions given by Eq. 14 are used to predict the optimum energy-pairs. The error propagation expression is used to determine the optimum energy-pair for each composite system to give the least uncertainty in our thickness, or density, measurements.

For the section that follows, all the experimental thickness, and density, measurements will be compared to the caliper measured thicknesses, and densities. All measurement deviations presented in the tables are determined by comparing the measured values to the values determined by a caliper. The deviations are given as percentages of the caliper value. Discussion of the results are presented in each section for the three composite studies.

6.2 Graphite-epoxy composite measurements

6.2.1 Optimization study

The prediction model for determining the optimum energy-pair uses the error-propagation expressions given in Eq. 14. To realistically predict the experimental uncertainties in the thickness measurements, we used experimental linear attenuation coefficients and experimental attenuation uncertainties given in Table 5.1. The beam transmission uncertainties are not present in our prediction models, because all our experimental counting statistics are high, on the order of several millions of counts. Therefore the beam transmission uncertainties are small and they can be ignored in Eq. 14.

The prediction model results for resin and fiber are plotted in Fig. 6.1 and Fig. 6.2, respectively. In the figures, a predicted uncertainty curve is plotted with a fixed low energy, E_1 , and varying the high energies, E_2 . Several predicted uncertainty curves are plotted with fixed E_1 to show the varying uncertainties with respect to different E_1 and E_2 energy combinations. The predicted uncertainties for the resin and fiber models are high for uncertainty curves having low E_1 values; i.e., uncertainty curves with E_1 values equal 13 keV and 15 keV. The predicted uncertainty curve reached a minimum for E_1 energy equaling 19 keV. Then the predicted values for the uncertainty curves increased with increasing E_1 values. There is an observable minimum in each uncertainty curve. The overall minimum uncertainty occurs at E_2 equals 31 keV for the uncertainty curve with fixed E_1 equaling 19 keV. This shows the optimum energy-pair for E_1 and E_2 to be 19 keV and 31 keV, respectively, for this graphite-epoxy composite study. The predicted uncertainties in the thickness measurements, from the optimum energy-pair, are 3.5% and 5.0% for the resin and fiber, respectively. All subsequent experimental measurements in this section will use this energy-pair.

6.2.2 Graphite-epoxy composite results and discussions

The experimental results from this study are shown in Table 6.1. The total effective thicknesses of resin in the samples varied from 0.200 cm to 0.493 cm, while the total effective thicknesses of IM-7 fibers remained the same at 0.254 cm. The fiber to resin ratios in the samples varied from 0.515 to 1.249.

Comparison between experimentally measured and caliper measured resin thicknesses are plotted in Fig. 6.3. The predicted uncertainties for the results in the figure are 3.5%. We see that the experimental measurements are in good agreement with the caliper measurements. Comparison between experimentally measured and caliper measured fiber thicknesses are

Figure 6.1. Predicted epoxy uncertainty using experimental linear attenuation coefficients and experimental attenuation coefficient uncertainties from Table 5.1. The thickness of the fiber and epoxy are 0.254 cm and 0.280 cm respectively

Figure 6.2. Predicted graphite fiber uncertainty using experimental linear attenuation coefficients and experimental attenuation coefficient uncertainties given in Table 5.1. The thickness of fiber and epoxy are 0.254 cm and 0.280 cm respectively

Table 6.1. Results of graphite composite measurements

Fiber-to-resin ratio	Thickness of epoxy-resin (cm)		Experimental deviation (%)	
	Caliper measured	X-ray measured	Epoxy-resin	IM fibers
1.249	0.203±0.001	0.203	-0.5	-0.7
		0.210	3.0	-3.2
		0.202	-0.8	1.1
1.025	0.248	0.250	1.0	-2.0
		0.251	1.3	0.1
		0.241	-2.6	4.5
		0.239	-3.6	5.7
0.989	0.257	0.252	-1.9	2.5
		0.269	4.9	8.4
		0.275	7.2	-12.8
0.908	0.271	0.273	-2.4	3.8
		0.277	-0.8	1.5
		0.283	1.4	-2.1
		0.284	1.7	-3.1
0.803	0.316	0.315	-0.5	2.7
		0.312	-1.4	2.2
		0.318	0.6	-0.7
0.704	0.361	0.362	0.2	1.2
		0.361	0.0	1.8
		0.358	-0.8	3.9
0.515	0.493	0.473	-3.9	9.4

Figure 6.3. Comparison between experimentally measured and known thickness of epoxy-resin (the uncertainties are derived from the error propagation analysis)

plotted in Fig. 6.4. The uncertainties for the results are 5.0%. The degree of data variation about the one-to-one correlation line is larger for the fiber than for the resin measurements. This is expected, because the attenuation of fiber, over the energy range chosen for this study, is smaller than that for the resin. Therefore, one expects a larger variation in the fiber thickness measurements [27].

In Fig. 6.5 we plot experimentally measured fiber-to-resin ratios against the fiber-to-resin ratios in our samples. Knowing the fiber-to-resin ratio is important in graphite-epoxy composites because the strength of the composite can be estimated from the fiber-to-resin ratio. The uncertainty for the fiber-to-resin ratios in the figure is 6.1%. Even though we are using extrapolated linear attenuation coefficients for IM-7 graphite fiber in this experiment, from the figure, we see that we can determine the fiber-to-resin ratio with good confidence. This technique can be improved for measuring the fiber-to-resin ratio if we can improve the accuracy of the linear attenuation coefficients at the upper and lower energies.

Comparison between experimental and predicted uncertainties displayed for fiber and epoxy-resin in Fig. 6.6 and Fig. 6.7, respectively, show that there is correlation in the uncertainty behavior; i.e., the uncertainties decrease when the energy difference increases, reach a minimum, then increase as the energy difference increases. These trough-shaped uncertainty curves result from the varied percentage uncertainties in the linear attenuation coefficients (see Table 5.1). The uncertainties in the linear attenuation coefficients are greater for those energy-pairs at 13 keV and 31 keV. Thus we see a rise in the predicted uncertainties at these energies, even when the energy differences are at their greatest. The error bars on the predicted uncertainty curve shows the variation in prediction due to a 0.5% variation in the linear attenuation coefficient uncertainties. For the dual energy combination of 19 keV and 31 keV, we found that the variation in the attenuation coefficient uncertainties of 0.5% can

Figure 6.4. Comparison between experimentally measured and known graphite fiber thickness (the uncertainties are derived from error propagation analysis)

Figure 6.5. Comparison between experimentally determined and actual fiber-to-resin ratios (the uncertainties are derived from the error propagation analysis)

Figure 6.6. Comparison between experimental and predicted uncertainty for graphite-epoxy fiber. The model uses experimentally measured linear attenuation coefficients and experimental attenuation coefficient uncertainties, given in Table 5.1. The model has ρ as 2×10^6 photon counts. The thicknesses of epoxy-resin and graphite fiber are 0.280 cm and 0.254 cm respectively

Figure 6.7. Comparison between experimental and predicted uncertainties for epoxy-resin. The model uses experimentally measured linear attenuation coefficients and experimental attenuation coefficient uncertainties, given in Table 5.1. The model has I_0 as 2×10^6 photon counts. The thicknesses of epoxy-resin and graphite fiber are 0.280 cm and 0.254 cm respectively

contribute up to 1.7% variation in the predicted uncertainty for the thickness measurements. This can explain the discrepancies between predicted and experimental uncertainties.

6.3 Bone-Plexiglas composite measurements

6.3.1 Optimization study

The prediction model for determining the optimum energy-pair uses experimental linear attenuation coefficients given in Table 5.3. The beam transmission uncertainties are not significant in our predictions. The uncertainties for the bone and Plexiglas linear attenuation coefficients are determined using the error-propagation analysis using Eq. 14. The uncertainties in the bone and Plexiglas attenuation coefficients are related to the thickness uncertainties in the caliper measurements used in the linear attenuation calculation. The uncertainties for the bone and Plexiglas coefficients are 1.0% and 0.3% respectively for all the energy measurements.

The predictions for determining the optimum energy-pair for bone and Plexiglas are shown in Fig. 6.8 and Fig. 6.9 respectively. The optimum energy-pair in the figures gives the least predicted percentage uncertainties. For this bone-Plexiglas composite system, the optimum energy-pair is 31 keV for the low-energy, E_1 , and 61 keV for the high-energy, E_2 . All subsequent experimental measurements in this section used this energy-pair.

From the two figures mentioned above, the predicted percentage uncertainty curves converge as the E_2 value increases. In fact, the predicted percentage bone uncertainties level off on the uncertainty curve with fixed low-energy value, E_1 , equaling 31 keV. This demonstrates that choosing energies that are farther apart than those energies used in this model will not significantly improve the uncertainty in the thickness measurements. Instead, to improve the experimental measurements, one should concentrate on improving the uncertainties in the linear attenuation coefficient measurements. We found the uncertainties in the linear attenuation coefficients strongly affect the accuracy of the thickness and density calculations [28].

Figure 6.8. Predicted bone uncertainty using experimental linear attenuation coefficients and experimental attenuation coefficient uncertainties given in Table 5.2. The thickness for bone and Plexiglas are 0.528 cm and 0.925 cm respectively

Figure 6.9. Predicted Plexiglas uncertainty using experimental linear attenuation coefficient and experimental attenuation coefficient uncertainties given in Table 5.2. The thickness for bone and Plexiglas are 0.528 cm and 0.925 cm respectively

6.3.2 Bone-Plexiglas composite results and discussion

The results from this study are shown in Table 6.2. The bone densities in the two sets of samples varied from 2.077 gm/cm^3 to 0.653 gm/cm^3 . The thicknesses of the Plexiglas in each set of the samples stayed the same. Comparison between experimentally measured and caliper measured bone thicknesses are plotted in Fig. 6.10. The uncertainty on the experimental data is 0.7% approximately. We see that the experimental results are in good agreement with expected values.

We measured the density of bone in our samples using Eq. 16 and Eq. 17. These results are shown in Table 6.3. Comparisons between experimentally determined, and simulated, bone densities are plotted in Fig. 6.11. The uncertainty for the density

Table 6.2. Experimental results for bone-Plexiglas composite

% Osteoporosis	Thickness of bone (cm)		Experimental deviation (%)	
	Caliper measured	X-ray measured	Bone	Plexiglas
a) Sample set 1				
0.0	0.808 ± 0.0013	0.811	0.4	2.5
20.4	0.643	0.647	0.7	1.1
27.9	0.582	0.585	0.6	1.1
34.6	0.528	0.528	0.0	1.5
b) Sample set 2				
0.0	2.4003 ± 0.0013	2.350	-2.1	2.7
34.6	1.569	1.641	4.6	1.1
46.9	1.274	1.363	7.0	-4.3
56.5	1.044	1.064	1.9	-2.2
68.6	0.754	0.771	2.2	-0.5
68.6	0.754	0.779	3.3	-7.0

Figure 6.10. Comparison between experimentally measured and known thickness of bone (the uncertainties are the size of the symbols; they are determined from error propagation analysis)

Figure 6.11. Comparison between experimentally measured and known bone density (the uncertainties are the size of the symbols; they are determined from the error propagation analysis)

measurements is 2.2% approximately. Again, we see good agreement in the measurements with expected values.

From the results given above, we have demonstrated in this study that we can accurately measure the bone density to 2.2% accuracy. Comparison between experimental and predicted uncertainties for bone and Plexiglas displayed in Fig. 6.12 and Fig. 6.13, respectively, show that they are in good agreement. The error bars on the predicted uncertainties show the variation of the prediction due to 0.5% variation in the attenuation coefficient uncertainties. This demonstrates that the error-propagation prediction model can successfully and accurately be used to predict the uncertainties in the thickness and density measurements for this bone-Plexiglas composite system. Thus we have shown promise in using this technique for detecting the onset of osteoporosis.

Table 6.3. Bone density measurements

% Osteoporosis	Bone density (gm/cm ³)		Experimental deviation (%)
	Simulated density	X-ray measured density	
a) sample set 1			
0.0	2.077±0.010	2.149	3.5
204	1.653	1.686	2.0
27.9	1.496	1.524	1.9
34.6	1.359	1.384	1.8
b) Sample set 2			
0.0	2.077±0.010	2.023	-2.6
34.6	1.358	1.432	5.4
46.9	1.102	1.142	3.6
56.5	0.903	0.905	0.2
68.6	0.653	0.640	-1.9
68.6	0.653	0.664	1.8

Figure 6.12. Comparison between experimental and predicted uncertainties for bone. The model uses experimentally measured linear attenuation coefficients and attenuation coefficient uncertainties given in Table 5.2. The thicknesses of bone and Plexiglas are 0.528 cm and 0.925 cm respectively

Figure 6.13. Comparison between experimental and predicted uncertainties for Plexiglas. The model uses experimentally measured linear attenuation coefficients and attenuation coefficient uncertainties given in Table 5.2. The thicknesses of bone and Plexiglas are 0.528 cm and 0.925 cm respectively

6.4 Aluminum-aluminum corrosion composite measurements

6.4.1 Optimization study

The prediction model for determining the optimum energy-pair for the corrosion study uses experimental linear attenuation coefficients and experimental attenuation coefficient uncertainties given in Table 5.3. The beam transmission uncertainties are not present in our predictions for reasons discussed above. The predicted uncertainty models for Al 2024 and Al(OH)₃ are shown in Fig. 6.14 and Fig. 6.15 respectively. For this corrosion study, the optimum energy-pair is 31 keV for the low energy, E_1 , and 61 keV for the high energy, E_2 . All subsequent experimental measurements in this section will use this energy-pair.

A similar observation is noted in this corrosion optimization study as was seen in the bone-Plexiglas optimization study. The predicted uncertainty curves converged as the value of E_2 increased. This means that choosing energies that are farther apart than those energies used in this model will not significantly improve the thickness, or density, measurements.

6.4.2 Aluminum-aluminum corrosion composite results and discussions

The results from this study are shown in Table 6.4. The percentage of material loss of aluminum 2024 varied from 0% to 50% in our samples. Comparison between experimentally measured and caliper measured aluminum 2024 thicknesses are displayed in Fig. 6.16. The predicted uncertainty for the measured aluminum 2024 in the figure is 3.1%. There is a systematic deviation in the aluminum 2024 results. Most of the measured aluminum 2024 thicknesses show a negative bias relative to the caliper thickness measurements. The average aluminum 2024 experimental bias is less than 4%, in close agreement with the predicted uncertainty of 3.1%. Thus we have shown that this technique can measure the material loss of aluminum 2024 with an accuracy of 4%. If this anomaly can be addressed, we predict that we can improve the current uncertainty of 4% to about 2% in the metal thickness measurements.

Figure 6.14. Predicted aluminum 2024 uncertainty using experimental linear attenuation coefficients and experimental attenuation coefficient uncertainties given in Table 5.3. The thicknesses of aluminum 2024 and aluminum hydroxide are 0.051 cm

Figure 6.15. Predicted aluminum hydroxide uncertainty using experimental linear attenuation coefficients and experimental attenuation coefficient uncertainties given in Table 5.3. The thicknesses of aluminum 2024 and aluminum hydroxide are 0.051 cm

Figure 6.16. Comparison between experimentally measured and known aluminum 2024 thicknesses (the uncertainties are determined from error propagation analysis)

Comparison between experimental and predicted uncertainties for aluminum 2024 displayed in Fig. 6.17 shows that they are in good agreement. This demonstrates that the error-propagation analysis can be successfully and accurately used to predict the uncertainties in the thickness measurements for aluminum 2024.

Comparison between experimentally measured aluminum hydroxide thicknesses and caliper measured thicknesses are plotted in Fig. 6.18. The predicted experimental uncertainty for aluminum hydroxide is 13.4%, while the experimental deviation for aluminum hydroxide varied from 0.1% to 211%. These large variation in the experiment may result from

Table 6.4. Experimental results of corrosion measurements

% Material Loss	Aluminum thickness (mils)		Experimental deviation (%)	
	Caliper measured	X-ray measured	Al 2024	Al(OH) ₃
0.0	40.00 ± 0.50	40.05 ± 1.45	1.2	
1.3	39.50	36.67	-7.1	200.0
3.8	38.50	38.07	-1.1	-211.0
5.5	37.80	36.34	-3.7	-105.0
6.5	37.40	36.41	-2.6	-81.8
6.5	37.40	36.28	-2.9	-75.5
10.0	36.00	35.50	-1.4	-143.0
10.0	36.00	34.38	-4.5	-20.9
10.0	36.00	34.73	-3.5	-28.7
10.0	36.00	34.42	-4.4	15.0
15.0	34.00	33.05	-2.8	0.1
15.0	34.00	33.73	-2.2	-20.4
23.8	30.50	29.62	-2.8	7.9
23.8	30.50	29.63	-2.8	0.4
26.3	29.50	29.28	-0.7	-20.5
36.3	25.50	25.40	-2.4	-34.6
36.3	25.50	24.88	-0.1	-17.0
38.8	24.50	23.57	-3.8	-17.3
50.0	20.00	18.08	-9.6	-3.1
50.0	20.00	18.51	-7.4	4.8

Figure 6.17. Comparison between experimental and predicted uncertainties for aluminum 2024. The model uses experimentally measured linear attenuation coefficients and experimental attenuation uncertainties given in Table 5.3. The thicknesses of aluminum 2024 and aluminum hydroxide are 0.091 cm and 0.014 cm respectively

Figure 6.18. Comparison between experimentally measured and known aluminum hydroxide thicknesses (the uncertainties are determined from error propagation analysis)

inconsistent packing of the aluminum hydroxide powder in the corrosion samples. This assumption is confirmed by Fig. 6.19, showing a comparison between experimental and predicted uncertainties for aluminum hydroxide. Here, we have a sample with a packed density for aluminum hydroxide that is different than the packed density for aluminum hydroxide used to measure the linear attenuation coefficient. Although we see a shift in the experimental uncertainties due to the density variation, the experimental uncertainty curve behaved as expected; i.e., the uncertainty decreases as the energy-pair moved further away from one another. In addition, the error bars on the predicted uncertainties show the variation of the prediction due to 0.5% variation in the linear attenuation coefficient uncertainties. This means that the difference in the thickness measurements cannot be accounted for by the linear attenuation uncertainty; instead, the difference is a result of the variations in the packed density for aluminum hydroxide.

The line drawn across the results in Table 6.4 indicates the FAA safety tolerance concerning material loss due to corrosion. Below this line are aluminum samples with unacceptable material loss of more than 10%. By observation, we see that this energy-dispersive technique gives uniform accuracy in the thickness measurements for Al 2024 for thicknesses ranging from 20 mils to 40 mils. Therefore we have successfully demonstrated that this x-ray technique can quantitatively measure the thickness of aluminum 2024 metal and detect the presence of its corrosion product - aluminum hydroxide.

Figure 6.19. Comparison between experimental and predicted uncertainties for aluminum hydroxide. The model uses experimentally measured linear attenuation coefficients and experimental attenuation uncertainties given in Table 5.3. The thicknesses of aluminum 2024 and aluminum hydroxide are 0.091 cm and 0.014 cm respectively

7. CONCLUSIONS

This thesis has been an experimental study of an energy-dispersive x-ray NDE technique for the purpose of material characterization of composite systems. Such information can be used to quantitatively determine material variation of each of the components in the composite which in turn can be used for estimating the material properties such as the strength of the composite and the densities of its components.

This study demonstrated the principle of the x-ray energy-dispersive technique for quantitative material characterization in composite systems. This x-ray technique worked well for determining the thicknesses and densities for composite components having the higher linear attenuation coefficient. For example, this technique accurately determined material thickness of epoxy-resin and aluminum-metal, and the density of bone, to 4% or less in each of the corresponding composite-systems. Looking at Eq. 7 we see that the product of the attenuation coefficient and the thickness enters the formulae. For an element that has a relatively large attenuation coefficient, a small change in the thickness will have the same effect on the result as a large change in the thickness of an element that has a relatively small attenuation coefficient. Therefore, the thickness measurements for the elements with lower linear attenuation coefficients were less accurate, in their respective composite systems.

The accuracy of this energy-dispersive technique is dictated by the uncertainties in the linear attenuation coefficient measurements. For the attenuation coefficients of solids, the uncertainty comes from the uncertainty of the caliper measurement; while for the powder, such as aluminum hydroxide, the uncertainty comes from the caliper measurement, and moreover from the powder-packed density in the vial. Therefore care must be taken in making caliper measurements for linear attenuation coefficient calculations.

The use of the germanium detector and multichannel analyzer provided high energy sensitivity, but certain obvious limitations are present, such as the inspection time. The inspection time for the current system is about a day for point-measurement. This fails to meet the demand for a rapid in-service NDE inspection technique. In addition, the experimental setup itself is a limitation, because this technique requires access to two opposite sides of the sample. And frequently, access to two sides is limited or impossible due to intervening structures. Although this x-ray technique presented in this study requires access to both sides of the composite samples, this study nevertheless gives us confidence to develop an x-ray backscatter technique that will require access to only one side of the sample. This will make in-service material characterization more practical.

The limitation for the immediate development of a rapid in-service inspection tool, using this x-ray technique, is the amplifier and the MCA systems. The amplifier and the MCA systems are the weakest link in the electronic system's throughput capabilities. The amplifier has a pulse-shaping dead-time of about 5 - 80 μ sec per pulse depending on the count rate. The MCA can have dead-time greater than 30% when count rate exceeds 30 kHz.

The need for the long inspection time is due to the need to acquire high counting statistics. The future of the research in this technique will be to replace the MCA with a single-channel analyzer (SCA); this will significantly improve the inspection time. In a two-component composite, we only need two energies to characterize the composite. Thus we only need two SCA to monitor the two energies. The energy sensitive response of the germanium detector can be gated using a SCA. By selecting an upper and lower voltage window, we can regulate the energy of x-ray photons monitored. The advantage that the SCA has over MCA is its high count rate. SCA can handle count rate upto 400 kHz. Thus it is possible to reduce the current inspection time by 50 times, approximately. From this, we can reduce the inspection time from 24 hours to half-an-hour. There is a drawback in using this SCA setup; the current

SCA system cannot perform the PUR subtraction on the signals. Though this can be overcome by constructing an electronic device that monitors the incoming signal for Pile-Up-Rejection.

Therefore in this thesis we have demonstrated this new x-ray energy-dispersive NDE technique is capable of characterizing material compositions in different composite systems with two components. We have also demonstrated that the error-propagation analysis can be used to predict the experimental thickness uncertainty. We have demonstrated with experimental results that error-propagation analysis can accurately predict the optimum energy pair and experimental uncertainty. Thus the analysis can be used as a preliminary study to evaluate the accuracy attainable with this technique before any experiment is performed.

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