Nondestructive X-ray methods for characterization of advanced aerospace materials

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Nondestructive X-ray methods for characterization of advanced aerospace materials

by

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To my father.
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1. INTRODUCTION

1.1 The Need for NDE in the Aircraft Industry

Advanced materials for use in the aerospace industry are presently being developed and applied at an astonishing rate. This pace is driven by the need for materials that can withstand higher operating temperatures and loads, yet remain cost competitive. The future in NDE is influenced by the increased performance demands on materials, while on the other hand, a new need has developed for inspection of old materials in aging aircraft. As the performance demands of aerospace materials push nearer and nearer the theoretical limit for strength, the allowed flaw size in traditional materials is driven smaller, making quality control more stringent. The promise of improved performance characteristics is also generating strong interest in other materials such as: exotic alloys, ceramics and reinforced composites. The last two issues involve increasing the performance of materials, but the aerospace industry is also going through a critical period in that many of the original commercial airliners are reaching their design-life limits. With natural resources becoming more and more limited, the cost of replacement is often prohibitive. The next decade will bring about many changes in the aerospace industry in that cost effective airworthiness inspection programs will be developed to extend the life of older aircraft.

To address the above issues, a parallel revolution is taking place in the area of nondestructive evaluation (NDE) to meet the needs of quality control groups inspecting these materials. Each of these materials brings with it a new set of inspection
requirements and subsequently techniques must be developed to meet those requirements. The designers are putting greater demands on quality control groups to improve NDE detection limits. These limits include finding 50 micron flaws (cracks) and quantifying the material characteristics. The aerospace industry historically is responsible for advanced material developments and is, therefore, concerned with flaw detection that will continue to push current nondestructive evaluation technology.

Even as the new techniques develop, several types of inspection will be used to perform material characterization and determine the airworthiness of these older fleets. Eddy currents have found success in detection of cracks that develop around fasteners on the skins of aircraft. X-rays are used to detect flaws in composite components as well as cracks and porosity in wrought and cast metallic parts. Ultrasonics is used extensively to inspect composites for delaminations and impact damage. The complexity of current airframes and engines will require all of these methods and more to safely predict life expectancy of an airplane. On the other end of the design process and for future designs, research in computer simulated inspection prior to manufacture will enable the designer to design inspectibility into a component to reduce both replacement and inspection costs later.

Several authors (5,13,19,25 and 26) have summarized the progress made in nondestructive evaluation of advanced materials, particularly composites, up to 1980. These advancements include ultrasonic attenuation and velocity measurements of void content, delaminations and thickness. Eddy current conductivity is being used for resin content quantification and dielectric tests for monitoring the cure of composites. X-rays are finding use in detecting porosity, foreign objects and cracks. Dye penetrant is another useful method for locating surface cracks, porosity and edge delaminations. The physics behind each of these methods has been fairly well understood for some
time, but recent instrumentation technology has made new applications of existing methods possible. An example of this was with the development of the digital oscilloscope used extensively in ultrasonic work. Similarly, new ultrasonic transducers are being designed to emit a specifically shaped wave pattern for quantitative flaw characterization.

From 1980 to the present, the trend in NDE is toward quantitative flaw detection. This means that flaws not only must be found, but sized and reconstructed as well. Many of the advancements are made possible through the use of the digital computer, such as: ultrasonic signal processing, X-ray image analysis, computer-aided tomography and in the collection of data from all the major NDE methods. The methods previously mentioned such as: eddy current, ultrasonic, X-ray, magnetic particle, liquid die penetrant and a host of others are all being refined for quantifying flaw measurements. Each of these methods has limitations and inspections may require more than one to detect certain flaws.

The following gives a summary of the uses for the most common NDE techniques. Eddy currents find widespread application in crack length determination on airframes such as around fasteners. Ultrasonics is used extensively for both production and in-service inspection of composite panels for delamination and porosity measurement. X-ray methods are used primarily during production for composite sandwich panel inspection and crack sizing, but portable generators are also being used in-service. The last two methods are becoming more and more quantitative as signal processing and image analysis techniques are developed. Ultrasonic and X-ray computed tomography (CT) are examples that are currently finding widespread usage in industrial applications. Only recently has X-ray tomography been refined for use in nondestructive evaluation of structural assemblies, while ultrasonic CT is only a gleam
Presently, in the area of quantitative X-ray techniques, much work has been done in image analysis of radiographs. Many manufacturers use full-size radiographs of components and highly experienced radiographers to find flaws of a critical nature. This type of inspection is dependent on the skill of the radiographer and can be very tedious. The most common quantitative method involves digitization of film negatives, since film is the most widely used detector today. The problem with this method is that the process is difficult to automate and is subject to errors if the original film image is not of good quality.

1.2 Review of Previous Work

The general problems facing the NDE community involve many techniques and a large number of people. Like any other technology achievements, the work done by others is the starting point for new developments. The work presented in this thesis involves characterization of advanced materials using a new digital X-ray detector and addresses problems common to several advanced materials. The problems include quantitative measurements of porosity and the quantitative measurement of material composition. A number of techniques for inspecting advanced materials have been developed in the last decade because of their increased usage. Many of the advancements being referenced are specific to one type of flaw or inspection procedure, but the techniques reported herein are just the foundation for a variety of applications.

In the area of quantitative porosity detection of advanced materials, the developments are limited. The most widely used nondestructive methods are volume and mass measurement or Archimede's method of weighing in water to calculate
density. In reinforced plastics, acid digestion, or high temperature decomposition are common destructive methods used. These techniques are subject to large errors and are also limited to small samples. Hsu, Nair, Rose and Uhl (8,9,10 and 18) have reported results on quantitative flaw detection and sizing on porosity in fiber reinforced composites using ultrasonic attenuation measurements. Roberts and Yuhas (23 and 33) also performed ultrasonic porosity measurements using a backscatter technique on the same composite samples used by Hsu. Those samples are studied in this thesis using an X-ray technique and compared to Hsu's results. The highest degree of success was found in samples with medium to low porosity levels, because the ultrasonic signal becomes highly attenuated in samples with high porosity levels.

Martin (13) reported limited success using an X-ray film technique for the measurement of resin content in graphite fiber reinforced epoxy composites. He used film densities to measure absorption coefficients of various samples and approximate resin content. The conclusion of his work is that film does not have the sensitivity necessary for detecting relative absorption variations in materials as complex as fiber reinforced composites. The work done by Martin revealed some fundamental problems with such measurements that must be addressed. The first difficulty can be labeled beam instability. He noted random X-ray generator voltage and current fluctuations, especially at low energies, that produce large errors in absorption coefficient measurements. This seems to be a common phenomenon with standard generators to date. The last two sources of difficulty that introduces significant systematic errors are a shift in the X-ray energy spectrum when a sample is placed in the beam and the inhomogeneity of advanced materials. The former is minimized if the sample thickness is kept small, but the latter is just a feature of these new materials.

Other problems that are encountered when inspecting light element materials such as
carbon fiber reinforced plastics (CFRP) are the energy dependence of absorption coefficients and the low flux of current generators at these energy levels. Wysnewski (32) studied the 10 - 50 kV energy range in an attempt to find optimal exposure levels for light elements such as CFRP. He measured the effects of air absorption and found it to be significant throughout this energy range and especially below 20 kV. Rudich et al. (24) developed a specialized high flux X-ray tube to be used for high-current operation at 6 - 25 kV ranges. This tube cuts inspection times by a factor of almost twenty. Inspection of lighter materials at lower energies takes advantage of the higher attenuation variations, therefore, increasing contrast and resolution to improve flaw detection dramatically.

The Northrop Corporation has incorporated an automated X-ray inspection system for inspection of F/A-18A composite assemblies that makes use of current detector technology. The instrumentation and technique as described by Murphy et al. (17) is a specialized application of the technique to follow. The use of a digital detector opens the door for computer reconstruction of flaws and a variety of other imaging techniques. The important point is that the well understood physical background for X-ray interaction with materials gives X-ray techniques for inspection a great deal of potential. The current limiting factor in tapping this potential is in technique development. Improvements can be made in generators and detectors, but the current ones can have immediate impact in next-generation nondestructive evaluation techniques.

The technique presented in this thesis is an example of a new application of existing technology, with scintillation detectors being one of the oldest detectors in use today. The use of these detectors for full image reconstruction scanning parallels work done in ultrasonics. Many systems, like the one at Northrop, use an array of detectors to speed
inspection times, but cost can become prohibitive for multiple detectors. A tradeoff must be made between inspection speed and equipment overhead. The technique detailed in the following section has distinct advantages in both these areas.

1.3 Scope of Research

The purpose of this thesis is to develop an automated X-ray technique for characterizing aerospace materials using a new digital detector. This technique can be used for process monitoring, material composition measurement, characterizing defects and monitoring these defects while in-service. The inspection method summarized in this thesis is tested on several advanced composites and ceramics to determine its suitability to quantify the porosity and or particle reinforcement content as well as variations due to heat treatment processing. The energy and material dependent interaction of X-rays with materials make it a natural tool for material characterization. The sensitivity to material variations is enhanced considerably, over film, with the use of a digital detector. This, when added to the high spatial resolution obtained by using a detector collimator, makes the digital detector a powerful feature mapping tool.

The material characterization technique presented herein uses the digital detector with a sample positioner to collect point by point intensity values that are necessary for calculation of linear absorption coefficients. This is done automatically with a program written for the IBM PC and requires little additional hardware over what a typical X-ray facility contains. The digital detector and supporting instrumentation is capable of measuring photon intensities and outputting photon counts. The advantage of such a detector is in its ability to be incorporated into an automated inspection procedure. An additional, and probably, more important advantage is the increased sensitivity over
other detectors such as film or fluoroscopy (image intensifier) systems. This sensitivity is necessary when measuring the small variations associated with defects such as porosity or resin content in CFRP or microcracks in ceramics.

The presentation that follows will document a study on material characterization using the digital (NaI) detector. The primary flaw being investigated is porosity in composite and ceramic materials, however, the technique is not limited to these materials. The levels of porosity in several samples is measured and preliminary work on porosity morphology is presented. An additional set of metal-matrix composite samples is studied that have variations due to heat treatment and particle reinforcement content. The samples chosen are used to test the technique and equipment and determine areas where errors become significant.

My initial efforts were spent learning the theory behind X-ray generation and interaction with materials as well as the analogous relations that describe ultrasonic and eddy current interactions. With this foundation, the next step was to fabricate, assemble and develop the equipment and procedures used for performing automated raster-scan inspections. This involved writing a Pascal computer code to control the positioners, detector instrumentation and data collection through a serial interface on an IBM-AT personal computer. I also designed and machined the collimators and positioner platforms used for positioning the sample. Once the equipment and procedure were established, verification testing was performed on the digital detector instrumentation. The last step was to apply the scanning inspection technique to a variety of materials.

The samples used for this study are carbon fiber reinforced composites, ceramics, particle reinforced metal-matrix composites and unidirectional ceramic fiber reinforced metal-matrix composites. The carbon fiber reinforced composites include crosswoven
carbon/PMR15 polyimide and unidirectional and quasi-isotropic graphite/epoxy. The
 ceramic samples are alumina (Al₂O₃), while the particle reinforced composites consist
 of SiC particles in a matrix of two aluminum alloys, 6061 and 7091. Lastly the ceramic
 fiber composites have unidirectional alumina (Al₂O₃) fibers in a magnesium matrix.

The necessary theory of X-ray interaction with materials is initially presented as
background for the calculations used in the results. The error estimation theory is
included to support the generation of error bars on the figures given in the results
section. The instrumentation, procedure and instrument verification testing is then
described, followed by the sample descriptions. Porosity measurements and metal-
matrix composite material characterization results are discussed as a means for verifying
the technique. Lastly, the conclusions and recommendations for future study are given.
2. THEORY FOR X-RAY INTERACTION

2.1 Introduction

In this section, the theory for monoenergetic and multienergetic X-ray interaction with a homogeneous isotropic material is presented. The sections to follow will describe this relation as applied to general composite materials and a simplified version that is used for relative density measurement. The last topic is error analysis in experimental measurement of relative density.

The interaction of X-rays with a homogeneous isotropic material is described in References (1,4,6,11,15,19,20 and 27) by Lambert's law:

\[ I(E) = I_0(E) e^{-\rho \mu(E)X} \]  

where \( I_0 \) and \( I \) are incident and transmitted beam intensity in front of and behind the object of interest respectively. These are functions of the energy of the beam that is controlled by the generator voltage setting. The other terms in the exponent are density, mass absorption coefficient and thickness of the material. The mass absorption coefficient is also a function of energy and is tabulated in References (14 and 15) for all the elements. This relation is the basis for the technique described in this thesis.

For the case of a monoenergetic beam, the three terms that are a function of energy become constants and Eqn. 1 is written as
The energy dependence of the mass absorption coefficient is not a simple relation and the common practice is to use what is known as the total mass absorption coefficient. This value is the summation of three absorption coefficients: coherent, incoherent and photoelectric, that are a result of the most common X-ray interactions with matter in the energy regime used for inspection. The references last mentioned give the details of these interactions, and McMaster et al. (14 and 15) gives the mass absorption coefficients in tabular and graphical form for various energies. Another way of quantifying absorption is with the linear absorption coefficient that is just the product of mass absorption coefficient and density ($\rho \mu$). This will be used extensively in the results to follow.

2.2 General Composite Materials

Now consider the general composite material as shown in Fig. 1a. An X-ray beam with incident intensity of $I_0$ passes through the material, resulting in a transmitted intensity of $I$. To model this interaction, a volume of the material is isolated in Fig. 1b. A cylinder is used since a collimated detector measures such a volume. Therefore, the volume probed by a collimated detector is $V=At$, where A is collimator aperture area and t is material thickness.
References (1, 4, 6, 12, 16, 20, 21 and 29) show that the interaction of a monoenergetic X-ray beam with such a material is written as

\[ I = I_o e^{-(\rho_1 \mu_1 \sum_{i=1}^{N} x_{i1} + \rho_2 \mu_2 \sum_{i=1}^{N} x_{i2} + \ldots + \rho_n \mu_n \sum_{i=1}^{N} x_{in})} \]  

(3)

where \( \rho \) is density, \( \mu \) is mass absorption coefficient and \( x \) is thickness for each of the materials. If the individual thickness contributions of each component are summed, Eqn. 3 becomes

\[ I = I_o e^{-(\rho_1 \mu_1 X_1 + \rho_2 \mu_2 X_2 + \ldots + \rho_n \mu_n X_n)} \]  

(4)

The above equations are the basis for many of the measurements to follow, because each material in a composite contributes to the overall attenuation. An important point
about the relations being used is that they are for a monoenergetic beam or a detector capable of detecting intensities in a specified energy window. The detector measures the integrated effect of all components in a composite material, but with some prior knowledge of the composition, the relative attenuation of certain components can be determined, Martin (13). Without specific component weight fraction information known, the general relations must be simplified using assumptions about material composition to perform feature measurements.

2.3 Carbon Fiber Reinforced Plastics

2.3.1 Relative density simplification

In the case of carbon fiber reinforced plastics (CFRP), such as in Fig. 2, certain assumptions can be made to simplify the general relation in Eqn. 3. Porosity measurement is facilitated using these assumptions. The general relation in Eqn. 3 is written as

\[ I = I_0 e^{-\left(\rho_F \mu_F X_F + \rho_R \mu_R X_R + \rho_V \mu_V X_V\right)} \]  

(5)

where F, R and V represent fiber, resin and void components. The relative densities and absorption coefficients between these three is a key point. Epoxy and several other standard resin systems consist primarily of carbon, oxygen, nitrogen and hydrogen. Therefore, to an X-ray beam, resin and fiber have very similar X-ray interaction behavior. However, air (or void) has a significantly lower linear absorption coefficient when compared to the other two. It is this difference that is used to measure porosity
by comparing the relative transmitted intensity variation between two samples.

Figure 2. A typical unidirectional fiber reinforced composite

Consider the two objects depicted in Fig. 3 in which a relative density difference is present.

Figure 3. Two general objects with different density and or mass absorption coefficient
Equation 2 gives the monoenergetic attenuation. Writing this equation for both objects in Fig. 3 and taking a ratio gives

\[
\frac{\rho_2 \mu_2}{\rho_1 \mu_1} = \frac{X_1 \xi}{X_2}
\]

(6)

where \( \xi \) is defined as

\[
\xi = \frac{\ln\left(\frac{I}{I_0,2}\right)}{\ln\left(\frac{I}{I_0,1}\right)}
\]

(7)

and the 1 and 2 denote objects 1 and 2. In this form, the relative linear absorption (\( \rho \mu \)) is compared for the two objects. Given one linear absorption coefficient, the other is calculated using Eqn. 6 and by measuring the thicknesses and intensities.

2.3.2 Absolute density relation

The density can be directly measured if a value of the mass absorption coefficient is known for the material of the sample. Equation 2 is solved to give

\[
\rho = -\frac{1}{\mu X} \ln\left(\frac{I}{I_0}\right)
\]

(8)

Just as density obeys the rule of mixtures for a composite material, so too does mass absorption coefficient. If prior knowledge of elemental composition is available, the absorption can be calculated from tabulated data (14,15). A subset from this reference is given in Table 1 and shown graphically in Fig. 4.
<table>
<thead>
<tr>
<th>Element</th>
<th>Density (gm/cm³)</th>
<th>Total Mass Absorption Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>10 KeV (cm²/gm)</td>
</tr>
<tr>
<td>Aluminum</td>
<td>2.702</td>
<td>26.39</td>
</tr>
<tr>
<td>Carbon</td>
<td>1.580</td>
<td>43.72</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>8.987(10⁻⁵)</td>
<td>0.3854</td>
</tr>
<tr>
<td>Iron</td>
<td>7.860</td>
<td>172.8</td>
</tr>
<tr>
<td>Lead</td>
<td>11.34</td>
<td>132.9</td>
</tr>
<tr>
<td>Magnesium</td>
<td>1.740</td>
<td>21.25</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>1.250(10⁻³)</td>
<td>3.699</td>
</tr>
<tr>
<td>Oxygen</td>
<td>1.429(10⁻³)</td>
<td>5.666</td>
</tr>
<tr>
<td>Silicon</td>
<td>2.330</td>
<td>34.43</td>
</tr>
<tr>
<td>Titanium</td>
<td>4.540</td>
<td>111.7</td>
</tr>
</tbody>
</table>
Figure 4. Common aerospace material linear absorption coefficients
2.4 Error Analysis

The results that will follow are all relative linear absorption coefficient comparisons. It is important that an understanding of the sensitivity of Eqn. 6 to each of the measured variables be established. The derivation that follows is one method for determining the error propagation effects.

Rewriting Eqn. 6 in the form

\[ \rho_2 \mu_2 = \rho_1 \mu_1 \frac{X_1 \xi}{X_2} \]  \hspace{1cm} (9)

makes it possible to vary each of the measurable or known quantities by a known percentage to quantify the resulting variation in \( (\rho_2 \mu_2) \). The linear terms: \( \rho_1, \mu_1, X_1 \) and \( X_2 \) are the simplest to predict. A percentage error in any of these values translates to the same error in the linear absorption coefficient.

The log terms are particularly interesting as they involve divisions within, and of the entire function as well. To quantify the effects of these terms, an iterative procedure was used to maximize and minimize Eqn. 7, where \( \xi \) represents the function made up of the log terms of interest. Base values for all the intensities in Eqn. 9 were chosen to match intensities commonly used in experimental measurements. Each of the four intensities was increased or decreased simultaneously by a known percentage to yield

\[ \xi_{\text{max}} = \xi + \delta \xi = \frac{\ln \left( \frac{I + \delta I}{I_o - \delta I_o} \right)}{\ln \left( \frac{I - \delta I}{I_o + \delta I_o} \right)} \]  \hspace{1cm} (10a)
\[ \xi_{\text{min}} = \xi - \delta\xi = \frac{\ln \left( \frac{I - \delta I}{I_0 + \delta I_0} \right)}{\ln \left( \frac{I_0 - \delta I_0}{I_0 + \delta I_0} \right)} \]  

(10b)

where \( \delta \) represents a variation in the base value.

This analysis resulted in the following observations as summarized in Table 2.

Table 2. Sensitivity results on the logarithmic terms in Equation 9

<table>
<thead>
<tr>
<th>Variation in Intensities, ( \delta I ) (% of counts)</th>
<th>Variation in Linear Absorption Coefficient, ( \delta(\rho_2\mu_2) ) (% of cm(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \pm 1 )</td>
<td>( \pm 12 )</td>
</tr>
<tr>
<td>( \pm 0.5 )</td>
<td>( \pm 8 )</td>
</tr>
<tr>
<td>( \pm 0.3 )</td>
<td>( \pm 4.35 )</td>
</tr>
<tr>
<td>( \pm 0.1 )</td>
<td>( \pm 1.45 )</td>
</tr>
</tbody>
</table>

This table shows the high degree of sensitivity that linear absorption has to variations in intensities. There is almost a 10 to 1 correlation, unlike all the other terms in Eqn. 9 which are 1 to 1. For this reason, it is very important that counting errors be minimized if accuracy is to be maintained in relative density measurements.

It has been shown in References (3,12,20 and 29) that digital detectors, such the NaI scintillation detector, can be statistically modelled using a Poisson distribution. This is a direct mathematical simplification of the binomial distribution under conditions that the success probability, \( p \) is small. In other words, the number of photons counted
by such detectors versus the number hitting the surface is a small percentage. For a Poisson distribution, the standard deviation about a mean count value is defined as

$$\text{S.D.} = \pm \sqrt{n}$$  \hspace{1cm} (11)

where \( n \) is the total number of photons counted by the detector in a given unit time.

This variance in the count values becomes very important as Table 2 shows. For this reason, care must be taken in choosing a high enough value for \( n \) so that the \( \delta I \) terms remain small. As an example of this effect, assume a total count value of 500,000 counts at a given collection location. The standard deviation is \( \pm 707 \) counts or 0.14\% of the total counts. From Table 2, this translates to approximately \( \pm 2 \)\% variation in the linear absorption base value. The choice of total counts must be weighed against the scan time limit allowed.

The errors associated with thickness and density measurements must not be forgotten as they too can become significant. In many cases, the surface roughness of a material can cause large variations. The means for arriving at a reference sample's density is also important. In many cases with plastic composites this is done using acid digestion. This technique is subject to large errors if not interpreted correctly.
3. INSTRUMENTATION AND SYSTEM VERIFICATION

3.1 Sodium Iodide Detector

The measurements made on samples described in chapter four have all been done using a digital sodium iodide detector coupled to a single channel analyzer. This detector is an inorganic scintillation detector that converts photon energy to visible light. The amount of light generated is proportional to the energy of the photon depositing that energy. A photomultiplier (PM) tube is coupled to the detector crystal to convert the weak light output into a corresponding electrical signal. The current pulse output of the PM tube is fed into a preamplifier that shapes the signal and an amplifier that boosts the amplitude in the form of a 0 to 10 volt pulse. The result is an energy sensitive detector response that can be gated using a single channel analyzer (SCA). Selection of an upper and lower voltage window regulates the energy of the X-ray photons monitored. The output from the SCA is sent to a counter/timer that counts pulses within a specified time period. Finally, the count values are downloaded to an IBM PC-AT via a buffered printout control/interface. The computer also controls the timer during automated scans. A flowchart of the detector instrumentation is shown in Figure 5 on the following page.

The digital detector is capable of collecting the intensity value within a selected energy window on a multienergetic beam (white spectrum). For most applications the energy window selected includes the entire spectrum (all energies) just as is the case for film. This ability of a digital detector to discretize the intensities throughout the spectrum and collect at a specified period in time is a powerful capability. Film is an
Figure 5. Digital (NaI) detector instrumentation
integration detector in that it collects photons of all energies for a given exposure period.

Scintillation detectors are widely used for measuring ionizing radiation and are one of the oldest techniques for doing so on record. The details on performance of such detectors can be found in References (1, 12, 20 and 29). In the detector saturation limits Section 3.6.1 that follows, experimental data are presented to quantify the saturation limits of the NaI detector. These limits must be avoided to maintain a linear response for the detector.

3.2 Collimators

The digital detector is placed within a lead shielding case that is fitted for interchangeable collimators. The shielding is of 0.25" lead as are the collimators. Figure 6 shows the design for the two collimators presently available. The purpose of these collimators is twofold. The first of these is to cut down on the number of photons allowed to reach the detector. These detectors are extremely sensitive, but they also have a finite saturation point, analogous to a funnel when filled too fast. The shielding is extended so as to enclose the entire NaI detector in order to eliminate unwanted scattered photons entering the detector. The second function of a collimator is to increase spatial resolution of the detector. The aperture size is shown in the system verification Section 3.6.2 to be the determining factor on image resolution quality. This becomes critical when doing flaw morphology measurements.

As part of this project, the collimator used for the results was designed and fabricated as shown in Fig. 6b. It consists of a single 0.25" lead plate with a 0.60 mm. diameter hole drilled in the center. The second collimator in Fig. 6a was also designed
Figure 6. Collimator designs: (a) dual aperture, (b) basic single aperture

* NOTE: All lead plate used is 0.25 inch thick
and fabricated specifically for this work and is a more flexible design. The slide-in assembly shown consists of two aperture plates held a fixed distance apart by the spacer. These plates are interchangeable to vary the size of aperture. The outer collimator case has a larger aperture to do initial beam collimation. The inner two apertures when aligned are used to allow only a finely collimated pencil-beam to reach the detector face. The plates have two degrees of freedom to ease the initial alignment. The spacer is present only to hold the plates a given distance apart. With proper plate selection, the collimator can allow a large or very small beam to reach the detector. The reason for such a design is to decrease aperture size for more detailed flaw characterization.

The geometry behind beam collimation is quite simple. The volume probed on an object placed between detector and source is a function of source size, collimator aperture and the distances from source to object and source to collimator. The values shown in Fig. 7 are typical for the scans performed in this study. The important point here is that the area surveyed on the object is for all practical purposes equal to the collimator aperture. The reason for this is due to the large distance from source to object and small distance from object to detector. However, these too can be varied to increase spatial resolution.

3.3 Positioners

An X-Y positioner is used to move samples during a Raster-scan inspection. The precision slides are manufactured by Daedal Inc. and are driven with stepper motors made by Compumotor Inc. The positioners are controlled with an IBM PC-AT using
$f_s$ = Generator Focal Spot Size (~10 μm)
$de$ = Collimator Aperture Diameter (~0.60 mm)
$do$ = Average Scan Diameter of Object (~580 μm)
$D_{so}$ = Distance from Source to Object (~36 in.)
$D_{od}$ = Distance from Object to Detector (~1 in.)
θ = Fan Angle (~0.02 deg.)

Figure 7. Geometry of object volume probed
two Compumotor PC-21 microprocessor-based indexer boards placed in the computer. These indexer boards send the control commands input by the user to the two motor/drives. The stepper motors are capable of one micron \((1 \times 10^{-6} \text{ m})\) steps and up to eight inches of total travel along both axes. The details of computer controlling the positioners is given in a Section 3.5 that follows.

3.4 X-Ray Source

The X-ray source used is a Ridge HOMX 160A microfocus generator with a 10 micron focal spot size approximately. The machine is capable of energies up to 160 KV and with beam currents of 2 mA or up to 5 mA of current at lower energies. It is important to note that any broad-beam or isotope source can be used and the microfocus capabilities are not being utilized for the study presented, indeed any radiation source can be used. The microfocus circuitry is actually disabled during scanning to stabilize beam intensity over time. All spatial resolution on the samples is obtained using detector collimation.

3.5 Controller Program

The experimental configuration is shown in Fig. 8 and is analogous to an ultrasonic through transmission (TTU) setup. The source and collimated detector are stationary, while the object is raster-scanned between. The scans can be either full two-
Figure 8. Raster-scanning equipment configuration
dimensional maps or one-dimensional line scans. All positioner movement and detector counting is controlled using an IBM PC-AT computer with a PASCAL program called "SCAN" to send the equipment commands. This program was developed as part of this thesis at the Center for NDE and allows an operator to interactively create a scan pattern tailored for both area necessary and length of time allowed.

The details behind the program are not included, but Fig. 9 shows a flowchart containing the logic. The user selects all scan parameters such as: type (1-D or 2-D), lengths and step sizes, count time at each position and other convenient options. The program then creates the ASCII commands that will be sent to the PC21 indexer board for positioning and to the TC588 buffered printout control/interface for counting, via the serial port on the PC. Once the count value is collected, the program then takes it from the TC588 and stores it on disk in a form for immediate graphical display.

3.6 System Verification

3.6.1 Detector Saturation Limits

The digital detector has counting limitations due to the efficiency of the crystal used as well as the preamplification and amplification circuitry coupled to the detector. The amplifier has adjustments for pulse peaking rate and shape that can be varied. The recommended setting for a NaI detector is the fastest available peak rate with a bipolar pulse shape. This was verified experimentally by varying peaking rates from 1, 3 and 6 μsec. along with pulse shapes from unipolar to bipolar. It was found that a 1 μsec. rate with bipolar shaping provides the highest count rates without saturation.

Saturation occurs when a detector and its supporting instrumentation (i.e., preamp. and amp.) can no longer keep up with the incoming flux of photons. The plot in Fig.
Input the scan parameters: i.e. 1 or 2-D area, step size, count time...

Calculate total time required and generate character strings needed to control positioners and detector instrumentation.

Send command to positioners to move.

Send command to detector to start counting.

Read in the total counts as sent from the detector.

Output the position and count data to file.

Send positioners the command to return to origin.

Figure 9. Flowchart of "SCAN" data acquisition program
10 shows experimental data that identifies the saturation limit of the detector used for this study. The current of the generator is varied for a given voltage setting to give a family of curves. The figure shows that linearity is no longer preserved above approximately 175,000 counts per second at high voltage. This limitation must be addressed for every scan. The speed of a scan is controlled by positioner speeds and count times. The only way to speed counting is to increase the efficiency of the detector and its instrumentation. The importance of high count values is given in Section 2.4 under error analysis.

3.6.2 Spatial Resolution

One of the most important features of the scanning technique presented in a following section is the high degree of spatial resolution. This is the ability of a detector to resolve small features in an object. The collimators described previously provide the resolution for the measurements in this study. The degree of resolution is tested experimentally using a standard X-ray resolution gage as shown in Fig. 11. The figure shows a 3X magnification image taken using the microfocus capabilities of the generator. Even though this is not a high magnification for a microfocus source (i.e. 100X), a point to note is the sharpness of the image. This gage is made of gold foil on lead with five lines cut through that come to a point. The point at which a detector, film or digital, loses the ability to distinguish between separate lines is the resolution limit.

The digital detector was tested by running a detailed line scan across different resolution levels of the gage until the lines were indistinguishable. These results demonstrated that the detectors resolution limit is controlled by the collimator aperture. The collimators used have circular apertures so to increase resolution, the hole can be decreased or angled with respect to the source. Angling the circular aperture produces a cateye shape with a narrower window in one direction as shown in Fig. 12.
1 μsec. peaking and bipolar amplification

Figure 10. NaI detector saturation test
Collimator

Source

Collimator

Circular Aperture

Cateye Aperture

Figure 12. Spatial resolution enhancement technique
A representative scan data set collected using a cateye aperture is given in Fig. 13.

The conversion from line pairs per mm. as used on the gage to distance on an object is quite simple. A line pair is defined as a gap plus a solid line. The resolution measurement is made using the distance from the outer peaks in Fig. 13, knowing that to be four and one half line pairs or nine lines. The result yields a resolution of 29 microns. It must be noted that this is not the limit of the detector, only the collimator aperture.
Figure 13. Spatial resolution measurement

X-ray resolution gage

17 lp/mm or 29 µm
0.25 mil step size
cat eye aperture
4. SAMPLE DESCRIPTION

4.1 Introduction

The features investigated in the materials examined fall into two main categories: porosity determination and general material composition characterization. The section that follows is separated in this way. Several material types have been studied and the relevant material properties supplied with these samples or measured at the Center for NDE are found in this section. The last samples described are used for flaw morphology measurements and contain well-defined flaws.

4.2 Porosity Samples

4.2.1 Graphite/polyimide composites

A set of graphite/polyimide or carbon/PMR15 samples to be more specific was provided by one of the industry sponsors to the Center for NDE. The set consists of ten coupons, each having six plies of 0/90 crosswoven with known amounts of porosity. The coupons are 1"x0.25" and typically 0.10" thick. These were fabricated specifically by the sponsor to contain varying amounts of porosity.

The panels from which each coupon was taken have been thoroughly studied using acid digestion by the sponsor and ultrasonic attenuation measurements at the Center for NDE, Hsu (8). The density has also been verified using mass and volume measurements, as the samples could not be destroyed. The surface texture is
nonuniform as is typical of crosswoven composites. The table that follows summarizes the known densities and void contents for each sample as provided by the sponsor or measured at the Center for NDE.

Table 3. Graphite/Polyimide (carbon/PMR15) sample set data as provided by the sponsor or measured at the Center for NDE

<table>
<thead>
<tr>
<th>Sample I.D.</th>
<th>Acid Dig.</th>
<th>Mass/Vol.</th>
<th>Volume %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(g/cm³)</td>
<td>(g/cm³)</td>
<td>Void Content</td>
</tr>
<tr>
<td>4</td>
<td>1.5342</td>
<td>1.5616</td>
<td>1.20</td>
</tr>
<tr>
<td>5</td>
<td>1.5222</td>
<td>1.5383</td>
<td>2.70</td>
</tr>
<tr>
<td>6</td>
<td>1.5026</td>
<td>1.5226</td>
<td>4.20</td>
</tr>
<tr>
<td>7</td>
<td>1.4980</td>
<td>1.5205</td>
<td>2.90</td>
</tr>
<tr>
<td>8</td>
<td>1.4543</td>
<td>1.4922</td>
<td>5.40</td>
</tr>
<tr>
<td>9</td>
<td>1.4002</td>
<td>1.4469</td>
<td>7.90</td>
</tr>
<tr>
<td>10</td>
<td>1.4050</td>
<td>1.4366</td>
<td>9.00</td>
</tr>
<tr>
<td>11</td>
<td>1.4215</td>
<td>1.4426</td>
<td>9.70</td>
</tr>
<tr>
<td>12</td>
<td>1.3852</td>
<td>1.3871</td>
<td>12.00</td>
</tr>
<tr>
<td>13</td>
<td>1.3929</td>
<td>1.3893</td>
<td>11.20</td>
</tr>
</tbody>
</table>

* a Acid digestion performed by the supplier on an adjacent area to where the samples were taken.

* b Measured using the sample dimensions and mass at Center for NDE.

* c Based on acid digestion results.

4.2.2 Graphite/epoxy composites

Two sets of graphite/epoxy samples were provided by Rohr Industries, an industry sponsor to the Center for NDE. The sets each consist of four coupons having sixteen plies with known amounts of porosity. The first set labeled with an A is unidirectional
and the second, labeled B, is quasi-isotropic. In this case quasi-isotropic refers to 
\([\pm45/0/90]_{2s}\) layup. The coupons are 1"x1" and typically 0.10" thick. These too were 
fabricated by the sponsor to give varying amounts of porosity. The surface texture of 
both sets is fairly uniform as is typical of composites fabricated with unidirectional 
tapes.

The panels from which each coupon was taken have been characterized using acid 
digestion by the sponsor and ultrasonic attenuation measurements at the Center for 
NDE, Hsu, Nair and Uhl (9,10). The table that follows summarizes the known 
densities and void contents for each sample as provided by the sponsor.

Table 4. Graphite/Epoxy sample set data as provided by the sponsor

<table>
<thead>
<tr>
<th>Sample I.D.</th>
<th>Density Acid Dig.(^a) (g/cm(^3))</th>
<th>Volume %(^b) Void Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unidirectional</td>
<td></td>
<td></td>
</tr>
<tr>
<td>A1</td>
<td>1.4880</td>
<td>6.51</td>
</tr>
<tr>
<td>A2</td>
<td>1.5576</td>
<td>2.04</td>
</tr>
<tr>
<td>A4</td>
<td>1.5804</td>
<td>1.14</td>
</tr>
<tr>
<td>A5</td>
<td>1.5901</td>
<td>0.20</td>
</tr>
<tr>
<td>Quasi-isotropic</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B1</td>
<td>1.5131</td>
<td>4.05</td>
</tr>
<tr>
<td>B2</td>
<td>1.5464</td>
<td>2.82</td>
</tr>
<tr>
<td>B4</td>
<td>1.5684</td>
<td>1.25</td>
</tr>
<tr>
<td>B5</td>
<td>1.5985</td>
<td>0.34</td>
</tr>
</tbody>
</table>

\(^a\)Acid digestion performed by the supplier on an adjacent area to where the samples 
were taken.

\(^b\)Based on acid digestion results.
4.2.3 Alumina ceramic

A set of alumina (Al₂O₃) samples was provided by Northwestern University. The specimens are made of Reynolds RCHPDBM alumina with a 3% PVB binder and pressed in a 1.75" die. The sintering temperatures were varied to produce varying amounts of porosity. The set consists of six samples that are 0.25" thick and 1.75" in diameter.

The densities for each coupon were measured at Northwestern using the Archimedes method, or in the case of the high porosities, by weight and dimensions. The density has also been verified using mass and volume measurements at the Center for NDE. The table that follows summarizes the known densities and void contents for each sample as provided by the supplier or measured at the Center for NDE. One note about these samples is that the thicknesses are highly uniform.

Table 5. Alumina ceramic (Al₂O₃) sample set data as provided by the supplier or measured at the Center for NDE

<table>
<thead>
<tr>
<th>Sample I.D.</th>
<th>Archimedes (g/cm³)</th>
<th>Mass/Vol. (g/cm³)</th>
<th>Volume % Void Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.9311</td>
<td>3.8740</td>
<td>1.38</td>
</tr>
<tr>
<td>2</td>
<td>3.8997</td>
<td>3.8400</td>
<td>2.16</td>
</tr>
<tr>
<td>3</td>
<td>3.8434</td>
<td>3.8109</td>
<td>3.58</td>
</tr>
<tr>
<td>4</td>
<td>3.6739</td>
<td>3.6362</td>
<td>7.83</td>
</tr>
<tr>
<td>5</td>
<td>3.3800*</td>
<td>3.3252</td>
<td>15.20</td>
</tr>
<tr>
<td>6</td>
<td>2.3000*</td>
<td>2.2802</td>
<td>43.20</td>
</tr>
</tbody>
</table>

*a Measured by the supplier using the Archimedes principle (weight in H₂O).

*b Measured using the sample dimensions and mass at Center for NDE.

*c Based on supplier's density results.

* Measured using mass and volume.
4.3 Composition Characterization

4.3.1 Alumina/magnesium composites

Two sets of unidirectional fiber-reinforced metal-matrix composites were provided by Westinghouse Research and Development Center, an industry sponsor to the Center for NDE. The fibers are made of alumina ceramic and the matrix is magnesium. The coupons have been heat treated using thermal cycling and isothermal treatment at New Mexico Tech. The material is (Al₂O₃)/Mg composite with 35% fiber volume fraction. One set was thermal cycled between 50 and 300°C from 500 to 2500 cycles. The other set was isothermally treated at 350°C from 20 to 480 hours. The cycled samples are 1.25"x0.50" and typically 0.10" thick. The surface texture on these is quite nonuniform due to the treatment process. The isothermally treated samples are typically 0.25"x0.50" and 0.25" thick. These have been cut without regard to fiber direction or parallel surfaces. The predominate feature present in these samples is microcracking as noted by the supplier. This cracking is attributed to the treatment process.

4.3.2 Aluminum/SiC composites

Three sets of SiC particle-reinforced metal-matrix composites were provided by Westinghouse R&D Center. The sets consist of bars of three aluminum alloys with varying amounts of SiC particle-reinforcement by volume. These bars are 0.5"x1.5" and typically 0.5" thick. The three alloys used are 2124, 6061 and 7091 aluminum. From this same lot, tensile specimens were machined to be tested when all forms of nondestructive characterization available at the Center for NDE have been exhausted. The purpose of these specimens is to characterize flaws which initiate failure. The
tensile specimens available are only 6061 and 7091 alloys. The nominal composition of 6061 aluminum is 1% Mg, 0.25% Cu, 0.6% Si, 0.25% Cr and 97.9% Al by weight. The composition of 7091 is unknown, but a typical 7000 series alloy has Zn as the main alloying element along with Mg, Cu and Cr. The dimensions of these specimens are 0.50"x1.50" gage section and 0.10" thick.

The samples have a variety of features present as summarized in Table 6. The primary feature is varying amounts of SiC particles by volume, but porosity and intermetallic compounds are also present. These intermetallic compounds are impurities such as manganese, iron, copper, chromium and titanium to name a few. The volume percentages of each component were determined using back-scattered-electron-image photographs. Image analysis was performed to determine the area percentages that in turn are converted to volume percentage. Westinghouse prepared a report to document internal ultrasonic and eddy current results on these samples.

These samples are particularly interesting because of the variety of features present. First, the matrix is an alloy that is composed of several elements along with aluminum. The samples also contain voids and intermetallic compounds. The elements in the intermetallic compounds are provided by Westinghouse, but the amount of each is unknown. These intermetallic elements are very important, since many have much higher linear attenuations than aluminum or SiC. The amounts of these heavy elements should be small if the alloying process is controlled, but the results will show that the 6061 samples are particularly dirty with these compounds. The digital detector's sensitivity makes it useful in sorting materials with unacceptable levels of these impurities.
Table 6. Aluminum metal-matrix (Al/SiC) tensile specimen sample set data as provided by Westinghouse

<table>
<thead>
<tr>
<th>Sample I.D.</th>
<th>SiC Particle Volume %</th>
<th>Inter. Met. Compound Volume %</th>
<th>Void Volume %</th>
</tr>
</thead>
<tbody>
<tr>
<td>7091 Aluminum Alloy</td>
<td>2730</td>
<td>0</td>
<td>6.9±2.6</td>
</tr>
<tr>
<td>2711</td>
<td>2.4±1.2</td>
<td>6.9±2.8</td>
<td>0.5±0.9</td>
</tr>
<tr>
<td>2712</td>
<td>2.3±1.5</td>
<td>4.4±2.6</td>
<td>0</td>
</tr>
<tr>
<td>2713</td>
<td>3.5±2.8</td>
<td>3.2±1.1</td>
<td>4.2±2.8</td>
</tr>
<tr>
<td>2665</td>
<td>3.7±2.1</td>
<td>6.9±2.8</td>
<td>1.6±1.4</td>
</tr>
<tr>
<td>6061 Aluminum Alloy</td>
<td>2045</td>
<td>0</td>
<td>5.2±2.2</td>
</tr>
<tr>
<td>2047</td>
<td>2.3±1.8</td>
<td>15.5±4.8</td>
<td>0</td>
</tr>
<tr>
<td>2099</td>
<td>2.6±1.7</td>
<td>2.9±2.2</td>
<td>0</td>
</tr>
<tr>
<td>2731</td>
<td>2.8±1.7</td>
<td>1.2±2.1</td>
<td>2.6±2.3</td>
</tr>
</tbody>
</table>

4.4 Flaw Morphology Samples

Two samples are used to measure flaw morphology. The first is the B1 Rohr unidirectional graphite/epoxy coupon. This was chosen because of its high void content and the tendency for voids to align with the fiber direction. The second sample is a machined cone in an aluminum plate that is 0.36" thick. The cone was produced using the tip of a drill bit so that the final cone dimensions are 0.09" deep and 0.375" in diameter at the base. Each of these was scanned over the entire surface to give a 2-D intensity map of the objects. This provides the necessary data to image features and also to size those features.
5. RESULTS AND DISCUSSION

5.1 Porosity Measurement

5.1.1 Introduction and procedure

The first application of the scanning technique introduced previously is for measurement of porosity in the carbon fiber-reinforced plastics and alumina ceramic samples described previously. The quantitative measurement of porosity is a difficult prospect, because of the many different types involved. With fiber-reinforced plastics, there can be interlaminar, intralaminar, spheroid, tunnel or needle-shaped porosity and even delaminations. All of these must be interpreted carefully and may be difficult to image with X-rays depending on the orientation. The photo that follows is a 50X optical photomicrograph of the graphite/polymide analyzed herein. Figure 14a is sample #4 and has 1.2% porosity and 14b is #13 with 11.2%. This illustrates the types of porosity and also the degree possible. Typically designers using such materials, plan the design allowables based on a 4% porosity level for secondary structural parts.

The control panels made for testing porosity measurements are fabricated in a variety of ways including: pressure variation or water introduction during layup and cure. These differences raise many questions such as: does the void or porosity replace resin, or does it increase thickness and hence displace the resin while leaving the total resin content a constant? This is an important issue since beam attenuation is a function of the thicknesses of materials it must pass through. To answer the above
Figure 14. 50X photomicrograph of crosswoven graphite/polyimide samples:

(a) sample #4 with 1.2% porosity, (b) sample #13 with 11.2% porosity
object thickness. The fiber and resin together are treated as one material due to their similar individual linear absorption coefficients. Porosity (or air) can be thought to either increase overall thickness or displace resin. Either effect is described using the relations outlined in Section 2.3.1.

The measurement of porosity in carbon fiber reinforced plastics is only made possible due to the similar linear absorption coefficients of carbon fibers and epoxy or polyimide resin at energies above 25 KeV. The mass absorption coefficients are practically identical throughout the entire energy regime. The linear absorption coefficient of air, however, is much less than either carbon or resin. Figure 15 illustrates this difference for the energies of the generator used in this study. The three curves are found using the composition of resin and density reported by Martin (13) for Narmco epoxy. The epoxy consists of ERL 0510b (tri-glycidyl-19H) epoxy resin with (15C, 1N, 4O, 19H) composition and 4,4' diamino-diphenol sulfone (Ciba Eporal) hardener with (2N, 1S, 2O, 16H, 21C) elemental composition. The density of this cured resin is 1.26 gm/cm³ and a typical carbon fiber is 2.0 gm/cm³.

A computer program named "XCOM" supplied by Martin J. Berger and J. H. Hubbell at the Center for Radiation Research under the National Bureau of Standards was used to generate the data in Fig. 15. This program uses a combination of theoretical calculations and experimental verification to output total mass absorption coefficients for elements, mixtures and compounds at different energies. The composition of the above resin and air are entered by volume percent, while pure carbon is used for the fiber curve. A density of 0.001185 gm/cm³ is used for air and a composition of 78.08% N, 20.95% O, 0.01% C and 0.96% Ar.

One point that must be addressed in doing a porosity measurement in carbon fiber
Figure 15. Carbon/epoxy and air linear absorption coefficients
reinforced plastic is whether the comparison of an effective linear absorption coefficient shows porosity, or merely variations in fiber/resin content. This point puts limits on what energies the porosity measurement can be made at, since the linear absorption coefficients are a function of energy. In order to lessen the effect of fiber/resin content variations on the porosity measurement, the energy used must be above 25 KeV. This restriction makes the linear absorption coefficients for resin and fiber practically the same, but significantly different from air.

The following table summarizes the results of a study to quantify the effects of porosity and fiber/resin variation on the linear absorption coefficient of the aforementioned Narmco resin in a carbon fiber reinforced system. The program from Berger is used to calculate the linear absorption coefficients for composites with the components mentioned above and having varying volume percentages of fiber, resin and porosity. The reference value is calculated assuming a fiber/resin content of 70/30 % respectively by volume and no porosity, a typical breakdown. The amount of fiber and resin was then increased and decreased by 5% without adding porosity and finally 2 and 0.5% porosity is added to the last case. The variations in fiber/resin content may be extreme, but this is being done as a worst case to get limits for the porosity measurement. An arbitrary high energy of 90 KeV is selected for the reason explained above.

The results shown in Table 7 show that porosity has a marked different effect on the linear absorption coefficient compared to fiber/resin variations. At porosity levels of 2%, the change in linear absorption is an order of magnitude greater than the change produced by 5% fiber/resin variations. The point at which porosity and fiber/resin variations are inseparable is near 0.5% porosity with 5% fiber/resin variations.
Table 7. Summary of the results of varying fiber/resin and porosity content on the expected total linear absorption coefficient of a CFRP material

<table>
<thead>
<tr>
<th>Fiber Volume %</th>
<th>Resin Volume %</th>
<th>Porosity Volume %</th>
<th>Lin. Absorp. Coefficient cm⁻¹</th>
<th>% Variation from Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td></td>
<td></td>
<td></td>
<td></td>
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Depending on whether fiber or resin content is higher, the linear absorption is practically equal to the case with no porosity. This is a limit of this measurement, but 1% porosity measurement is quite acceptable in most applications. Therefore, small variations in fiber/resin content will not have a drastic effect in the porosity measurement. This also helps to address a previously mentioned question of whether porosity displaces or replaces resin, because the standard way of measuring material
variation is by using volume percentages.

Porosity measurement is made much easier in the ceramic material studied, because the base material is primarily homogeneous unlike CFRP. This also applies for materials like cast aluminum or iron as long as impurities are minimal. The alumina samples provided excellent results because the thickness is extremely uniform.

The procedure used to characterize the materials in this study is analogous to ultrasonic through transmission (TTU) techniques. The object of interest is scanned between the stationary source and collimated detector. The total photon count or intensity is collected for each position and later reduced to give a linear absorption coefficient. The relation in Eqn. 6 of the theory section is used to obtain the relative linear absorption coefficient. With the absorption coefficients known, the volume percent porosity is given by

$$C_2 = \left(1 - \frac{\rho_2 \mu_2}{\rho_1 \mu_1}\right) 100 + C_1$$

where $C_1$ and $C_2$ are the porosity levels in samples #1 and #2 respectively. The density in sample #1 (the reference sample) must be determined externally, such as with acid digestion or Archimede's principle. The relation in Eqn. 12 is not the standard porosity equation, since the mass absorption coefficients are present. An assumption is used to simplify Eqn. 12 by making the two mass absorption coefficients equal. This is a reasonable assumption since the base composition of a CFRP is fairly well controlled and the mass absorption coefficient is a function of the material components. The effect of porosity on the overall mass absorption coefficient are minimal compared to the effect of porosity on overall density. Therefore, Eqn. 12 simplifies to
In terms of experimentally measured quantities, Eqn. 12 is written as

\[ C_2 = \left( 1 - \frac{\rho_2}{\rho_1} \right) 100 + C_1 \]  

(13)

by substitution from Eqn. 6.

The linear absorption measurement requires that the incident and final intensities be collected at each point. This is very difficult since two detector would be required and physical interference becomes a problem. The incident intensity should be a constant over time, since it is only a function of generator settings and distance from the source. However, experience has shown that generators are not stable over time.

To correct for the instability of the generator, the scans used in this study begin off the sample, pass over and end off the sample. This gives two data sets for incident intensity that are averaged to correct for beam instability and one final intensity set over the sample of interest. As part of this thesis, a Pascal program was written to analyze these data sets. The program reads the data file, selects the range to be statistically analyzed, as input by the user, and returns the average and variance for that subset. A program was also written to analyze any subset of a two-dimensional dataset.

5.1.2 Graphite/polyimide composites

Two typical line scan data sets collected for a porosity measurement are shown in Figs. 16 and 17. These plots are repeat scans across the graphite/polyimide sample #6.
Figure 16. Graphite/polyimide sample 6 initial scan
Figure 17. Graphite/polyimide sample 6 repeat scan
The two upper peaks are incident intensity off the sample and the lower is the final intensity behind the sample. The important features are the statistical variance in the incident intensity as described by Eqn. 11 and the feature dependent signal on the sample. The incident intensity is relatively constant as expected for a stable generator or for short scan times. The features on the sample are real as seen in the repeat scan of Fig. 17 that also illustrates the spatial resolution of the detector. The sample is shifted in the x-direction because the positioner started at a slightly different initial point. The two plots demonstrate the repeatability of the technique.

The figures that follow show the results of the X-ray technique for measuring porosity. The results in Fig. 18 are four separate scans of each carbon/PMR15 sample plotted with the average and acid digestion results. The energy used was 25 kV and 1 mA for a 5 second total count time at each position on the samples. The four scans are actually two sets at two different locations on the samples. Each line scan at a location is repeated for system checking and improved statistical analysis. Sample #4 is used as the reference since it has the lowest porosity level.

The variability is attributed to the spatial sensitivity of the detector and the interpretation of the analyst in selecting a representative subset of data for calculating the necessary intensities. The data reduction program, previously described, relies on the user to select endpoints for the data subsets used in generating incident and final intensity values for each sample. The error bars are found using the relations outlined in Section 2.4 and take into account the errors in thickness measurement and intensity counting. A thickness measurement error of 0.5 % is used throughout the results since the caliper used has that level of precision. The counting error is calculated using the variance in incident intensity for each scan data set.
Figure 18. Graphite/polyimide X-ray measurement results
Figure 19 shows the X-ray results plotted against other techniques. The ultrasonic data are from Hsu (8) on the same samples, while the classical density and thickness results are taken from the measured densities and thicknesses of each sample. Thickness is often used as a rough estimate of porosity content. The comparison between these results is quite good, although it must be noted that porosity is variable from point to point and none of the results are from the exact same locations. Therefore, some scatter is expected, but the X-ray technique is one of the most spatially sensitive, due to the fine collimation being used.

5.1.3 Graphite/epoxy composites

The next four plots show the graphite/epoxy composite results in the same form as Figs. 18 and 19. Figure 20 gives the X-ray results for the unidirectional graphite/epoxy samples from Rohr Industries and Fig. 21 compares that to other techniques as reported by Hsu and Nair (9). The energy used was 30 kV and 0.5 mA for a 3 second total count time at each position. The next two figures repeat these results for the quasi-isotropic samples of the same material. The image analysis results presented in Figs. 21 and 23 are from Hsu and Uhl (10) using photomicrographs.

5.1.4 Alumina ceramic

The last of the porosity results in Fig. 24 is of the alumina ceramics and cannot be compared to other NDE techniques due to data limitations for these samples. The energy used was 55 kV and 0.5 mA for a 2 second total count time at each position. The reference line is from density measurements taken at Northwestern University. The correlation to the X-ray technique is very good. This is attributed to the uniformity of sample thickness and homogeneity of the material.
Figure 19. Graphite/polyimide technique comparison results
Figure 20. Unidirectional graphite/epoxy X-ray results
Figure 21. Unidirectional graphite/epoxy technique comparison results
Figure 22. Quasi-isotropic graphite/epoxy X-ray results
Figure 23. Quasi-isotropic graphite/epoxy technique comparison results
Figure 24. Alumina (Al₂O₃) ceramic X-ray results
5.2 Materials Characterization

The second major application of the scanning technique is in determining material variations due to processing. The results are divided into two groups: heat treatment and particle reinforcement content. Both sets of samples are from Westinghouse Inc. and both are metal-matrix composites. The first set involves two different heat treatment processes on alumina/Mg fiber reinforced composites. Figure 25 shows the linear absorption coefficient verses the number of cycles between 50 and 300°C. The energy used was 55 kV and 0.5 mA for a 2 second total count time at each position on the samples. The average of two scans is shown and a decrease with the number of cycles is seen. This may be due to the increased microcracking and delaminations reported by Westinghouse as cycle numbers increase. The linear absorption coefficient would change, depending on the orientation of the micro-cracking. The increase or decrease depends on the angle of cracking relative to the incident beam. The more the cracking is oriented parallel to the beam, the greater the decrease in absorption.

The data in Fig. 26 show the variations in linear absorption coefficient with number of hours of isothermal treatment. The energy used was 55 kV and 0.5 mA for a 2 second total count time at each position on the samples. A significant drop in the absorption coefficient is seen up to fifty hours and then it remains relatively constant. The two plots may not be compared directly since the two sample sets are not from the same original lot. The point marked with a star on Fig. 26 represents the untreated sample from Fig. 25. There is a possibility that the two sets are not from the same lot, so this point may not be valid for the isothermal results. The point to be made here is that a trend is seen in both sets and they support the predictions of the suppliers.
Figure 25. Thermal cycled metal-matrix composite X-ray results
Figure 26. Isothermally treated metal-matrix composite X-ray results

Unidirectional Metal-matrix (Al₂O₃/Mg)

X-Ray Average
X-Ray scan 1
X-Ray scan 2
Untreated sample (possibly a different lot)

Number of hours at 350°C
The last three figures summarize the results for the SiC particle reinforced aluminum tensile specimens from Westinghouse. Figure 27 shows a linear correlation between SiC particle volume percent and linear absorption coefficient for the 7091 base alloy samples as is expected. However, the next figure is not as well behaved. The energy used was 55 kV and 0.5 mA for a 2 second total count time at each position on the samples. The 6061 alloy samples have a combination of effects that are seen in Fig. 28. The linear absorption at 20 % SiC content is quite high, but independent inspection using eddy currents at Westinghouse supports this result.

The reason for the increase on the one 6061 sample is due to the presence of intermetallic compounds made from elements such as titanium, iron and chromium bonded to aluminum and silicon. These compounds come from impurities in the alloying process and all would increase absorption relative to aluminum. Several of the samples also have porosity levels that must also be accounted for. Figure 29 is corrected for these effects in the case of the 6061 samples.

The method used to correct for the intermetallics involves calculating the linear absorption for these compounds at the energy used in Fig. 27. The energy used is 55 kV, therefore the linear absorptions are taken at 37 kV or at two-thirds the maximum energy as a general rule-of-thumb. Since the composition of these compounds is not known, an assumption that they are composed of equal parts of titanium, iron, chromium, aluminum and silicon is being made. This may be inaccurate since the first three heavy elements are probably found in smaller amounts than the aluminum and silicon. This is because the aluminum and silicon are true alloying components. The region shown in Fig. 29 gives the maximum and minimum linear absorption of the 6061 samples after correcting for the intermetallics. The reason for the band is that the
Figure 27. Al/SiC metal–matrix tensile specimen X-ray results
Figure 28. Al/SiC metal-matrix tensile specimen X-ray results
Figure 29. Corrected Al/SiC metal-matrix tensile specimen X-ray results
Westinghouse report has error bars on the volume percent composition of intermetallics and these are used to give the region that is shaded. The width of this band is a function of the uncertainty in the composition of the intermetallics.

As a check of the measurement, a reference point is added to Figs. 27 and 29. The star represents a measurement made on a plate of 6061 shop aluminum. The assumption here is that no porosity or intermetallics are present. This is useful since the linear absorption shown in all the results is an effective value for the entire energy spectrum of the generator. To compare to tabulated absorption coefficients, a weighted average must be made, the standard approximation is to use the absorption value at two-thirds of the maximum energy. Measuring this reference point experimentally performs the verification without having to calculate a weighted absorption or look at one energy window with the detector instrumentation and compare directly to tabulated values.

5.3 Flaw Morphology

The last results presented are used to illustrate the imaging abilities of the NaI detector in a scanning configuration. The first image shown in Fig. 30 is of a conical flaw in an aluminum plate. The energy used was 60 kV and 1 mA for a 3 second total count time at each position. The image is seen in topographic view in Fig. 31 where the contours of intensity are shown. This representation makes sizing of the flaw quite easy using the digitizing capabilities of the software package that the data is displayed with. The following figure is a more realistic example as applied to the unidirectional graphite/epoxy Rohr sample A1. The energy used was 20 kV and 1 mA for a 3 second
Figure 30. Machined cone flaw in a aluminum plate
Figure 31. Cone flaw topographic contour plot
total count time at each position on the sample. The scan in Fig. 32 was taken part on
and part off the sample and the signal to the right is from a tunnel void. The porosity in
unidirectional composites generally follows the fibers and the scan was able to map
these features. These figures illustrate the spatial resolution and potential for flaw
reconstruction that the digital detector possesses.
Figure 32. Tunnel voids in unidirectional graphite/epoxy
6. CONCLUSIONS

This thesis has been an experimental study of the X-Ray characterization of advanced aerospace materials. A digital (NaI) detector in a raster-scanning configuration is used to inspect carbon fiber reinforced plastics, ceramics and metal-matrix composites for porosity and material variations due to processing. A technique for measuring the relative linear absorption coefficient is presented and used to calculate volume percent porosity levels. An additional set of results is included on flaw morphology using the two-dimensional mapping ability of the raster-scan technique.

The use of the digital detector in the configuration shown is relatively new. Such a technique has great potential since the detector and collimator provide high sensitivity and spatial resolution. Certain obvious limitations are present such as the difficulty in inspecting an in-service assembly. X-ray equipment is portable, but access to both sides of the assembly is necessary. In many instances this is possible and especially in quality control inspection of parts prior to assembly.

The work presented has used the entire spectrum of the X-ray beam for the results shown. This spectrum and the energy dependence of the mass absorption coefficient is a potentially powerful tool. The detector used for this study has a sodium iodide crystal, but germanium detectors provide better energy resolution at the low energies common to CFRP inspection. This feature when coupled with a multi-channel analyzer (MCA) may make the absorption coefficient measurements reported by Martin (13) possible.

The errors associated with the porosity measurements are within useful limits if certain care is taken in collecting the data. The counting statistic variance can be
minimized by increasing total counts. This increases scan times unless a higher flux source is used. Such sources are either currently available or can be made with relatively minor expense and modification to existing hardware. The sensitivity of these detectors makes thickness measurements very important. A simple laser setup using interference patterns is presently available for doing very accurate thickness measurement. The beam instability problem can be dealt with by using a second detector to monitor the incident intensity throughout a scan. The intensity fluctuations of the generator will then be normalized for every point on the object. This has the advantage of using existing generators without extensive circuit modifications to stabilize voltage and current fluctuations.

The high spatial resolution of the collimated detector is shown in the results section. This resolution makes flaw reconstruction possible as demonstrated on the tunnel voids in unidirectional graphite/epoxy. The potential of such a technique is fairly obvious, but the applications are as yet unknown. Tomography is the next logical step for a feature mapping technique like the one shown herein. For the majority of cases, however, objects needing inspection have simple geometries with a large amount of flat or slightly curved surfaces. Therefore, the expense and time needed for a full tomography setup and inspection is not warranted.

Based on the results presented in this study, it is concluded that the use of a digital detector for measuring material variations is a viable technique. It should be noted that the method is not restricted to a microfocus generator or composite materials. Such materials present the greatest difficulty due to their inherent inhomogeneity. The potential of this technique will only be enhanced with the use of other digital detectors and a multi-channel analyzer.
7. REFERENCES


