X-RAY MEASUREMENT OF MATERIAL PROPERTIES IN COMPOSITES

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INTRODUCTION

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. 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. This demand for improved performance characteristics is also generating strong interest in other materials such as: exotic alloys, ceramics and reinforced composites. A need exists for characterizing these advanced materials for composition variations, flaw content, inclusions and porosity using nondestructive techniques at all stages of the materials life cycle. These stages include initial characterization of a new material, process control during the manufacturing of the material, quality control of incoming material, and the in service inspection of the final part.

The importance of quantitative measurement techniques is illustrated by porosity. The detection of porosity in ceramic composites is not sufficient, since good ceramic composites have a level of porosity easily detected. Knowledge of the amount of porosity is necessary to adequately characterize the quality of the material. The use of many techniques will be required for the adequate determination of the quality of these new materials. Ultrasonic, eddy current, thermal techniques, have been widely reported(1,2,3). One powerful technique for preforming these types of inspections that until recently(4,5) has been unsuccessful is the use of X-rays. Although the early results(6) using film based detection methods were unsuccessful due to insufficient sensitivity of the film, the use of scintillation detectors give excellent sensitivity and subsequently excellent results. In this paper we will illustrate the utility of an X-ray based technique for the quantitative determination of the levels of porosity in several classes of materials, metal matrix fiber, re-enforcement content, and the detection of process control problems in metal matrix composites, and the determination of the morphology of the flaw.

Instrumentation and Samples

The experimental configuration consists of a stationary X-ray source and a scintillation detector(4). The detector is shielded to protect the detector from the scattered radiation and is collimated to define a pencil beam of radiation (Fig. 1). The size of the collimation for our experiments is typically 0.6mm in diameter and is adjustable as
The equipment consists of a computer controlled x-z sample positioner and NaI scintillation detector, an X-ray source, and radiation shielding. The measurement is performed in two to five minutes resulting in the volume percent of the porosity.

needed to both larger or smaller sizes. The sample is moved through the X-ray pencil beam by a computer controlled sample positioner, it is this step interval together with the size of the collimation control the spatial resolution and the scan speed. We have measured spatial resolutions, without special effort, of thirty microns. Any X-ray source including isotope, microfocus, and high energy generators can be used as a source. Some care must be taken with the degree of collimation and the flux output of an X-ray generator since the scintillation detector does have a saturation limit.

The materials studied include carbon-plastic composites, ceramics, and metal matrix composites. These samples represent a wide variety of materials, inspection problems, flaws types, and, further, represent a wide range of material processes. Carbon-plastic composites, in particular, represent a large collection of inspection problems, specifically the presence of porosity, the uniformity of the resulting material, and the need to know the morphology of the flaws. The metal matrix composites represent the need for process control in that the amount of reinforcement in the metal, and identifying whether porosity is present must all be monitored. Ceramic samples illustrate the need for sample uniformity and the need to measure the level of porosity or the amount of densification and the presence of inclusion. Each of these materials illustrate some of the types of problems that the X-ray technique reported here is capable of measuring and characterizing.

In the example of the carbon plastic composite there are several pieces of information that can be obtained, namely, the level of the porosity, the flaw morphology, and most importantly whether the indication is the result of fiber resin separation or porosity. Figure 2 illustrates the measurement of porosity by several different methods including ultrasonic, radiographic, acid digestion and volumetric density measurements in carbon plastic composites. All values are from the same
samples except for those indicated for the acid digestion. These are from coupons that were adjacent to the samples upon which the NDE measurements were done. As can be seen the, all of the techniques track the porosity levels in the set of samples. There are several points that can be made concerning the X-ray measurements. The large variability in the porosity is not due to limitations of the X-ray technique but rather to the extremely fine spatial resolution of this technique. The spatial resolution of the detector at 30 microns is much smaller than an individual bubble, so at one point the composite will have the normal properties while at another point the detector may be looking at the bubble. This feature leads to the ability to map the detailed morphology of a flaw and is illustrated in Fig. 3. This sample is a unidirectional graphite-epoxy composite. It is well known the bubbles in this type of composite form long tunnel shaped voids along the fiber direction. The beginnings of one of these voids easily seen. Although the scans that map the flaw morphology can take a lot of time, the detailed information obtained can allow the identification of the flaw formation process much easier. This type of procedure is most valuable in process control stage of the development of new materials.

The third piece of information that the X-ray method can obtain is a determination of whether the variation in the signal is due to a local variation in the material properties, in this case the fiber-resin ratio or whether it is due to a void. This capability is one of the few NDE methods able to measure porosity in carbon plastic composites. The test for whether a variation in the X-ray intensities is due to a fiber-resin separation, due to porosity, or to some combination of the two is determined by making two measurements at different energies. The first measurement is at low energy, say between 10 and 30 keV. If an intensity anomaly is found, the determination of whether porosity contributes to this anomaly is done with a second measurement at a high energy, say 100 keV.

A summary of the volume percent porosity results obtained from five different methods giving excellent agreement. The measurements were done for each technique on the same set of samples which were made with various amounts of porosity.
Fig. 3. The generation of a two dimensional scan enables the morphology of a flaw to be shown. The sample is a unidirectional graphite epoxy composite and shows the presence of tunnel voids running along the fiber direction.

keV. If the size of the intensity variation is the same at the high energy, then the source is porosity. Examination in Fig. 4 of the energy dependent X-ray absorption coefficients for air, graphite fibers, and epoxy resin show that at energies above 100 keV the difference between the fiber absorption coefficient and the resin absorption coefficient is very small, while there is a difference of orders of magnitude with that of air at this energy. At high energies where the difference between the fiber and resin coefficients is very small, any variation in the fiber-resin ratio will have very little effect on the transmitted X-ray intensities. The resulting error in a measure of porosity that would occur if caused by a fiber-resin variation of 5% from its nominal value is 0.5%. This is well within the limits of the uncertainty of ±1% in the porosity measurement.

The separation of the effects of a combination of porosity and fiber-resin separation is more difficult in that at the higher energy the intensity is reduced in proportion to the ratio due to each cause. For this situation both measurements at the high and low energies are needed to determine the proper proportion. Measuring the fiber-resin ratio is possible with this technique, however, it is a difficult experimental measurement to perform.

The measurements illustrated with the carbon-plastic composites can easily be done on other materials. The results shown in Fig. 5 are porosity measurements on alumina ceramic. The porosity levels vary from a few percent to forty per cent by volume. This technique, as can be seen, is applicable to a much larger range of porosities than ultrasonics or the archimedes method can measure. These methods have problems due to the connectivity of the pores in samples with high porosity. The displacement of the liquid in the archimedes method and the entry of the ultrasonic coupling media lead to poor results when the porosity levels get above 20% to 30%. Finally, the X-ray technique can measure, as shown
The liner absorption coefficient for graphite, and epoxy resin, and air are plotted as a function of energy. Note that at energies above 100 keV the difference in the coefficients of the fiber and the resin are very small, while the coefficient for air is very small. At 20 keV there is a large difference between the fiber and resin absorption coefficient.

The porosity in alumina ceramics is shown for a range of porosities between 1% and 40%.
in Fig. 3, the density variations in the material as a function of the position. This measurement is not equivalent to a slice image obtained from a tomographic scan. As with any projection technique, the depth information is lost, however, the integrated uniformity of the projection data often yields sufficient information in less time than the tomographic scan time.

The last example of the materials that we have studied is metal matrix composites. In Fig. 6 we show a calibration curve for the linear absorption coefficient as a function of the volume percent of the reinforcement material. In this case the matrix material is aluminum 7091 alloy with particle SiC reinforcement. In a second case with aluminum 6061 alloy with SiC particle reinforcement the processing control of the material for the case of 20% SiC of the particle concentrations failed. The presence of intermetallic compounds, porosity, and the incorrect amounts of particle gave a very large discrepancy in the X-ray attenuation measurements. These results agree with those of an eddy current(7,8) and ultrasonic measurement on these samples.

![Graph](image)

**Fig. 6.** The linear absorption coefficient for metal matrix composites of aluminum with SiC particle reinforcement is plotted against the particle percentage present. The dashed lines are the aluminum alloy is 6061, the solid line is the 7091 alloy.

**CONCLUSIONS**

We have presented an X-ray method for determining material properties in a wide range of materials. This technique is reasonably fast and is certainly inexpensive. The results are comparable or better than the measurements by other techniques, at least in the examples where data for both techniques are available. This method, using the energy sensitivity of the NaI detector, is able to distinguish the difference between porosity and material concentration variation. The high spatial resolution, on the order of 30 microns, give the ability to map detailed flaw morphology. The results reported here make this technique a strong candidate for an inspection process in a variety of areas. The
implementation of the energy dependent X-ray measurements to determine the material segregation, for example the fiber-resin ratio in graphite composites, and the application to new materials represent ongoing work.

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REFERENCES

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