

X-RAY MEASUREMENT AND POROSITY IN GRAPHITE/POLYIMIDE COMPOSITES

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INTRODUCTION

One of the persistent problems in the control of quality in materials used in structures is that of porosity. Whether the material is a casting, an epoxy composite, or a complex ceramic or metal matrix composite, the presence of porosity, depending upon the severity, causes a degradation of the parts performance characteristics and their longevity. The detection of porosity in many industries is important in two ways, namely, in material quality control and in monitoring process control (1). One class of materials where both of these problems arise is the graphite composites (2-4). To date, the inspection of these composites using an X-ray method has been unsatisfactory in that the film methods frequently tried did not have the sensitivity resolution to yield a quantitative measure of the porosity volume percent. Such a measure becomes even more critical in ceramic composites in that the difference between a good material and a poor one is a level of porosity rather than the presence of porosity, as is the case in graphite epoxy composites. We wish to report results of an X-ray method to measure porosity in materials using the graphite polyimide composites as an example. The method is certainly not restricted to this type of graphite composite or indeed to graphite composites.

EXPERIMENTAL APPARATUS

The equipment used consists of any X-ray or gamma ray source, a x-y sample positioner, a NaI scintillation detector coupled into a photon counting circuit, a collimator, and a PC computer controller. The counting circuit consists of a high voltage supply for the photomultiplier tube, an amplifier, a single or multichannel analyzer, a counter-timer, and a buffered interface to the computer. The scintillation detector and the positioner are controlled by the PC computer. This automated data acquisition allows large quantities of data to be collected and immediately analyzed. The arrangement of the equipment can be seen in Figure 1. In this configuration the source and detector are stationary and the sample moves through the beam. There are several features that are important to consider. The beam of radiation is collimated at the detector and as with any collimated beam, the alignment is important. In this system the size of the beam allowed into the detector affects both the time required for data collection and the spatial resolution of the volume interrogated. If the beam

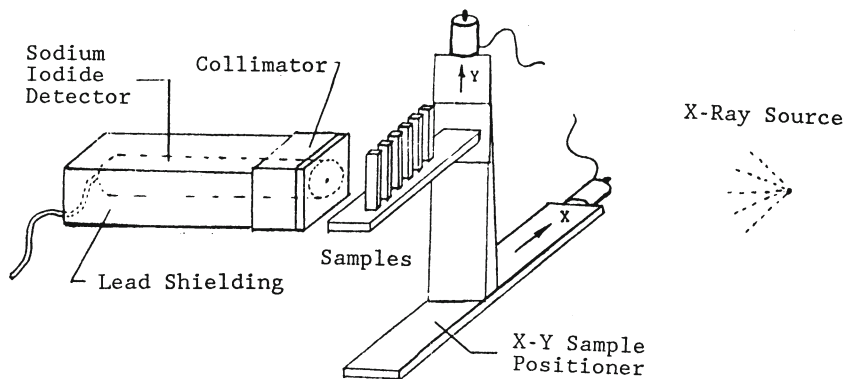


Fig. 1. The equipment consists of a computer controlled x-z sample positioner and NaI scintillation detector, an X-ray source, and radiation shielding. The measurement is performed in two to five minutes resulting in the volume percent of the porosity.

originates from a typical radiography source and is not collimated at the source, a radiation shield will be required to protect the scintillation detector from scattered radiation. The shielding enclosure used in our setup is 0.25" lead completely surrounding the detector. The collimation is done by placing a hole of 0.6mm diameter in the front face of the radiation shielding. Data collected from this system are shown in Figure 2. The samples used were a set made with varying degrees of porosity. Optical micrographs (5) of the samples are shown in Figure 3. These photographs show the different types of porosity that can typically occur in graphite composites. The measurement of this porosity can be done in an absolute or relative mode. The data shown in Figure 2 are relative measurements. Two samples, where one has a known porosity, are placed on the positioner. The measure of the porosity is obtained by measuring the differences in the absorption between the standard and test samples. There is one point of ambiguity in the conversion of the absorption difference to a volume percent porosity. Absorption differences between the resin and the fibers in any composite can compete with the absorption difference that occurs with a void. The linear absorption coefficient of polyimide at 50 kev, a typical energy at which the data are taken, is 0.30 cm^{-1} , while the linear absorption coefficient for graphite fibers at the same energy is 0.294 cm^{-1} . This is compared to that null absorption of a void. While it is possible to construct an example of an extreme segregation where there is very poor mixing of the fiber and resin that could mimic the magnitude of the difference in absorption observed for porosity, it is unlikely. This is indicated by the fact that typical fiber volume fractions for graphite composites are known to a few percent. An air gap is inserted to separate the samples. The air gaps before, in between, and after the samples are used to ensure that the variations in the signals are not due to time dependent beam fluctuations. This is a serious problem that must be addressed in that, in many radiography X-ray generators, the intensity can be unstable. This is especially so during the warm up period. It is not usually such a severe problem with film, since the sensitivity of film is much less than that of a scintillation detector. The signature of a stable beam intensity is the same number of the photon counts in each of the air gaps. The relative measurement of porosity rests on the attenuation of the X-ray flux and is expressed quantitatively in equation 1.

$$\frac{\rho'}{\rho} = \frac{x' \ln(I'/I'_0)}{x \ln(I/I_0)} \quad (1)$$

where the primed quantities refer to the reference sample, rho is the material density, x is the X-ray path length through the sample, I_0 is the initial photon intensity, and I is the final photon intensity.

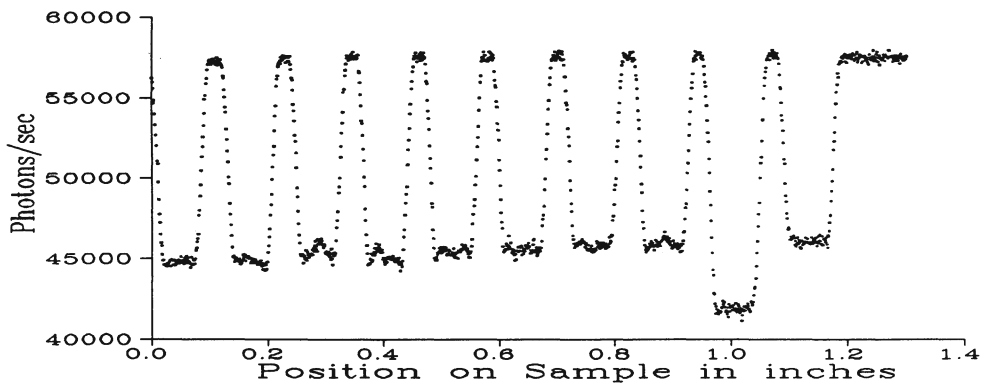


Fig. 2. A typical data set consisting of several samples of varying thickness and porosity. The physical thickness of the sample must be normalized. Note the stability of the source as represented in the air gaps.

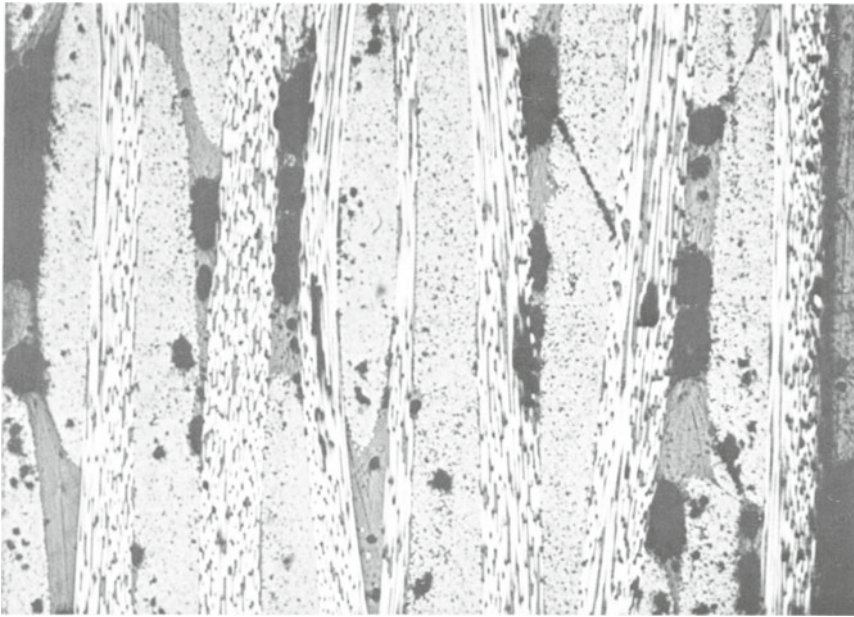


Fig. 3. An optical micrograph of a sample for which the X-ray measurements were done. Note the different kinds of porosity in the resin and in the fiber bundles.

The effect of the reference sample is to eliminate the need to know the energy dependent absorption coefficient. This is not the case in an absolute measurement, where the detailed knowledge of the energy distribution of the initial photon intensity and the material absorption coefficients are required. This indicates that the relative measurement, if a characterized sample is available, is the easier to implement. Indeed with industrial x-ray generators one has to deal with a complex white x-ray spectrum, making the calculation of the absorption effects very difficult. This is the reason for the use of an energy sensitive detector. The NaI scintillation detector when used with a single channel analyzer can select out the desired energy, greatly simplifying the resulting data analysis. When using the scintillation detector in this mode, extra care must be taken to avoid detector saturation. The entire spectrum is incident on the detector, however, only the photons of the selected energy are being counted. The maximum of the bremsstrahlung spectrum is at two thirds of the maximum energy. We should note that we have had success using the weighted average of the intensity and absorption coefficients without a serious loss of accuracy in the final measurement. It should be further noted that with the equipment described for this measurement the necessary measurements to determine the initial energy spectrum and the absorption coefficients can be made.

The results of measurements on ten samples manufactured with varying degrees of porosity are presented in Figure 4. The degree of porosity in this set of samples ranges from one percent to thirteen percent, as measured by acid digestion of neighboring portions of the panel. Plotted with the X-ray and acid digestion results are measurements of the porosity from ultrasonic attenuation (6) and those obtained from a classic dimension measurement, weighing, and subsequent

computation of the percent porosity. As can be seen, the results from the various techniques are in good agreement.

RESOLUTION AND SENSITIVITY

This system for measuring porosity has a great deal of flexibility concerning the type of measurement desired in that the volume inspected (the spatial resolution of the scan), the sensitivity, and the time of the scan can be tailored to the operators needs. Two features control the spatial resolution, namely, the size of the aperture and the step interval of the sample positioner. The parameters affecting the sensitivity of the measurement include the intensity of the X-ray or

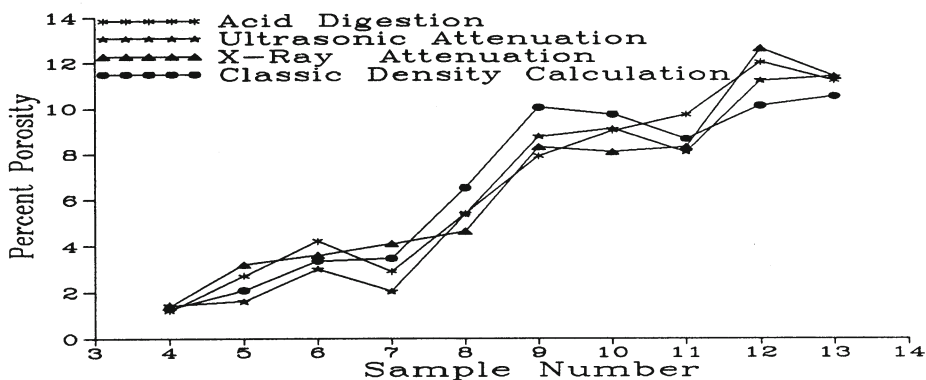


Fig. 4. A summary of the volume percent porosity results obtained from five different methods giving excellent agreement. The measurements were done for each technique on the same set of samples which were made with various amounts of porosity.

gamma ray source, the distance that the detector is from the source (radial divergence of the beam), and the size of the aperture. The time for one scan is controlled by the photon intensity at the detector and the number of step intervals in the scan. For most commercial X-ray generator sources care must be taken to avoid saturating the detector. The aperture used in these measurements is a 0.6 mm. diameter hole, one quarter of an inch deep and when used with a two mill step interval, the system has a spacial resolution of thirty microns. This resolution is limited by the size of the aperture as is indicated in Figure 5. This level of spatial detail gives the possibility of doing very fine morphological studies on different types of flaws in most any material. Indeed, knowledge describing the shape of existing flaws can be very

telling about the way that the flaw was formed. This in turn enables the process control to be modified. By using a large aperture one measures the average attenuation over a larger volume.

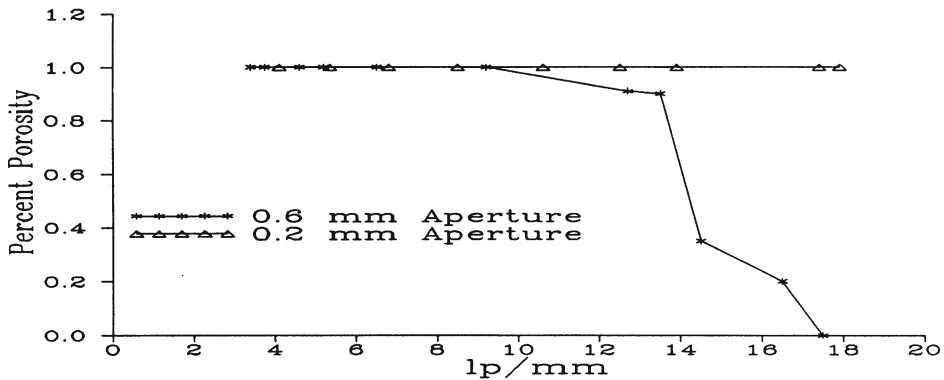


Fig. 5. The spatial resolution of the detector is limited by the size of the collimator aperture. As seen from the plot, the spatial resolution for the smallest aperture is better than 30 microns. A resolution of 1 indicates that the separation between lines is clear and sharp.

The sensitivity to variations in the material density of this technique is controlled by the fluctuations in the photon counts and as this noise is governed by Poisson statistics, the noise level is given by the square root of the number of counts collected. For one percent sensitivity in material variations in the sample to be measured, the counting noise must be significantly less. Counting rates of fifty thousand counts per data point will give one half of a percent random noise contribution to the measurement. This condition, together with the number of steps in the scan, control the time for data acquisition. For the measurements of porosity shown in Figure 2 the data collection time was three minutes. A computer program analyzes the data as it is collected and computes the relative volume percent of the porosity.

CONCLUSIONS

We have demonstrated the successful use of an X-ray inspection method for the quantitative measurement of porosity in polyimide graphite composites. This method, using a digital detector interfaced into a computer, has measured porosity levels down to one percent with an accuracy of plus-minus one half percent. The cost of this system, if one has a source of penetrating radiation, is approximately twelve thousand dollars, and includes the detector, positioner and PC computer. The time for the collection and analysis for such a measurement was three minutes. The measurements compare with the results of two independent measures of the volume content of voids, in particular ultrasonic attenuation and dimensional measurements to calculate the density. This method is applicable to any material that can be penetrated by the X-ray or gamma ray with sufficient quantities to satisfy the count statistics. In principle any application inspecting for volumetric type flaws that uses a through transmission ultrasonic technique could be a candidate for inspection with this X-ray method. Among the many applications that conform to this limitation are material qualification, process control in the fabrication of materials, and any in service components that have the requisite access. A number of these applications, namely the characterization of the morphology of voids and the application of this technique to other materials such as aluminum casting, ceramic components, are under consideration for immediate attention in our facility. We plan to report these results in the near future.

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