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TITLE X-RAY GAUGE MEASURES AREAL DENSITY VARIATIONS AS  
SMALL AS 0.1%

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# X-Ray Gauge Measures Areal Density

Variations as Small as 0.1%

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## ABSTRACT

A gauge measures areal density variations as small as 0.1% in low density plastic foams and other materials. It has a spatial resolution of 0.5 mm. This system is safe, portable, easy to use, and is readily automated. The 6 keV x-ray flux from a  $^{55}\text{Fe}$  source is collimated to a 0.5 mm diameter beam and attenuated by the sample. Transmitted x-rays are detected with a proportional gas detector and counted with a scaler-timer. After measuring the x-ray attenuation in two areas of the sample, the areal density variation is calculated using an expression derived from  $I/I_0 = \exp(-\rho x)$  where  $I$  is the transmitted flux,  $I_0$  is the incident flux,  $\rho$  is the mass attenuation coefficient of the sample,  $\rho$  is the foam density,  $x$  is the thickness of the sample, and the product  $\rho x = D$  is the areal density. Measurements sensitive to 1% and 0.1% areal density variations take three minutes and four hours per point, respectively, in  $0.05 \text{ g/cm}^3$  plastics, 1 cm thick. Absolute areal density is

also measured to  $\pm 3\%$  using existing x-ray attenuation tables. However, better accuracy may be obtained by calibrating the system with known samples.

## X-Ray Gauge Measures Areal Density

Variations as Small as 0.1%

### INTRODUCTION

A gauge measures areal density variations as small as 0.1% in low density plastic foams and other materials. If the linear dimensions are known, the density variation of these materials can be calculated. With a spatial resolution of 0.5 mm, this system is safe, portable, easy to use, and is readily automated. The 6 keV x-ray flux from a  $^{55}\text{Fe}$  source is collimated to a 0.5 mm diameter beam and attenuated by the sample. Transmitted x-rays are detected with a proportional gas detector and counted with a scaler-timer. After measuring the x-ray attenuation in two areas of the sample, the areal density variation is calculated using an expression derived from  $I/I_0 = \exp(-\mu x)$  where  $I$  is the transmitted flux,  $I_0$  is the incident flux,  $\mu$  is the mass attenuation coefficient of the sample,  $\rho$  is the foam density,  $x$  is the thickness of the sample, and the product  $\mu x = D$  is the areal density. Measurements sensitive to 1% and 0.1% areal density variations take three minutes and four hours per point, respectively, for  $0.05 \text{ g/cm}^3$  plastics, 1 cm thick. Absolute areal density is also measured to  $\pm 3\%$  using existing x-ray attenuation tables. However, better accuracy can be obtained by calibrating the system with known samples.

Low density plastic foams with densities in the range of 0.030 to 0.050 g/cm<sup>3</sup> are used in inertial confinement fusion targets where the areal density variations in the foam are required to be less than 0.1% ( $\Delta D/D \leq 0.001$ ), where  $\Delta D$  is the variation in areal density and, in this case,  $D$  is the mean areal density. Furthermore, the areal density must be measured over areas as small as (0.5 mm)<sup>2</sup>. These foams are literally as light as a feather and have nearly the same floating properties as feathers when they are dropped. They are also quite fragile, and it is difficult to measure their linear dimensions with a mechanical device without damaging the sample. Furthermore, the requirement to measure areal density over (0.5 mm)<sup>2</sup> areas precludes weighing samples of this size as this would destroy the larger sample which are, typically, 1 cm by 4 cm by 1 cm thick.

The absorption of the 6 keV x-ray lines from a <sup>55</sup>Fe source is an ideal way to measure the areal density variation of this material because it is a completely non-destructive technique. The basis for density and thickness measurements by x-ray absorption was published by Roentgen<sup>1</sup> in 1898. More complete descriptions of the technique are given in refs. 2-4. As applied to target fabrication for inertial confinement fusion, x-ray absorption is used in refs 5-9. In this paper a radioactive source, rather than an x-ray generator, is used to make an x-ray beam. The source makes this method much simpler, easier to use, and allows the apparatus to be easily transported from laboratory to laboratory, while maintaining very high precision.

## APPARATUS

The gauge is shown schematically in Fig. 1. An x-ray beam is formed by collimating the flux from a  $^{55}\text{Fe}$  source with two collimators. The sample is mounted on a two-axis stage which moves the sample perpendicular to the collimated beam. Photons in the transmitted x-ray beam are detected by a xenon-methane gas proportional detector ( $\text{CO}_2$  - Xe would work as well) whose output signal is amplified and routed to a timer-scaler. The timer-scaler will count one pulse from each photon detected in a pre-set time. To measure a spatial variation in the areal density, the sample is placed in the beam and the total number of transmitted photons,  $I(1)$ , is measured for a set time,  $t$ . Then another part of the sample is illuminated by the x-ray beam, and the number of transmitted photons,  $I(2)$ , is measured in time,  $t$ . The difference in the areal density,  $\Delta D$ , between the two locations is given by

$$\Delta D = -\frac{1}{u} \ln \left( \frac{I(2)}{I(1)} \right).$$

However, the statistical uncertainty in the measurement,  $\delta(\Delta D)$ , is given by

$$\delta(\Delta D) = \frac{2}{u} \frac{\delta I(1)}{I(1)}$$

where  $\delta I(1)$  is the statistical uncertainty in  $I(1)$ , and where  $I(1) \approx I(2)$  and  $\delta I(1) \approx \delta I(2)$  are assumed.

To measure  $\delta I/I$  smaller than 0.1%, it is essential to mount the gauge on a rigid frame in a vibration-free area so that there is no relative motion between the source and the detector that will change the measurement of the photon flux. It is also necessary to limit the count rate in the detector to 500 counts/sec or less to minimize electronic pile up of the pulses in the electronics. Higher rates may be used with more sophisticated electronics which use pile-up rejection techniques. Typical background rates are 1 count/sec.

The  $^{55}\text{Fe}$  source radiates at only two x-ray energies, the energies of the Mn  $K_\alpha$  and  $K_\beta$  lines at 5.89 keV and 6.49 keV, respectively. For the measurements presented here, this source is treated as a monoenergetic source, which greatly simplifies the analysis. Furthermore, the 2.6 year half-life of  $^{55}\text{Fe}$  means that the intensity of the source, which decreases by less than 0.1% each day, will vary in a known way. Therefore, there is no need to monitor variations in the incident x-ray flux as one needs to do with an x-ray generator. Other radioactive sources are available if higher energy x-rays are needed for thicker or more dense materials. Hence, this method may be used with other materials.

Even though the proportional gas detector cannot resolve the  $K_\alpha$  and  $K_\beta$  lines of the  $^{55}\text{Fe}$  source, this detector and the timer-scaler provide an accurate way to measure the x-ray attenuation. The performance of the gas counter and timer-scaler was compared to the performance of a Si(Li) detector used with a multi-channel analyzer. The Si(Li) detector easily resolves the two K lines. However, the timer-scaler measured the

attenuation more accurately than either of the K peaks in the multi-channel analyzer. Because the timer-scaler sums the entire energy spectrum above the background noise, it has larger statistics and, hence, a smaller standard deviation than the K peaks. For the same acquisition time, the standard deviation of fifteen measurements made with the timer scaler was smaller than the standard deviation of either of the K peaks. Of course, summing all the channels in the multi-channel analyzer above the background noise is equivalent to using the timer-scaler, and these sums agreed with the sums from the timer-scaler. Therefore, it is not necessary to resolve the  $K_{\alpha}$  and  $K_{\beta}$  peaks with a more expensive Si(Li) detector and multi-channel analyzer.

## RESULTS

This x-ray gauge accurately measures areal density variations as small as 0.1%. A 1  $\mu\text{m}$  thick polystyrene film was mounted on 1 mm of polyethylene, and the transmitted flux was measured through 1) the polyethylene and 2) the polyethylene with the polystyrene film, a 0.1% areal density change. To hasten these measurements by increasing the transmitted flux, two 0.25 cm diameter collimators were used rather than the 0.5 mm collimators. The areal density of the polystyrene film is  $2.3 \times 10^{-4} \text{ g/cm}^2$  with a standard deviation of  $0.6 \times 10^{-4} \text{ g/cm}^2$  as determined by the x-ray gauge. This value is within two standard deviations of  $1.2 \times 10^{-4} \text{ g/cm}^2$  as measured with an alpha particle energy loss spectrometer over a 0.65 cm diameter area and  $1 \times 10^{-4} \text{ g/cm}^2$  as measured with transmission interferometry. In both of the latter two measurements,



the polystyrene foil was measured by itself, freestanding. Neither of these latter two measurements are usable with the polystyrene mounted on the 1 mm of polyethylene.

To demonstrate that this gauge has adequate sensitivity with the 0.5 mm diameter collimators, measurements of the incident beam intensity through these smaller collimators were made over four days and corrected for the decay of the source. Fifteen measurements of about 72,000 counts each have a standard deviation of 165, which is 0.6 of the square root of 72,000. Hence, the uncertainty in the measured transmission through the sample is limited by only the counting statistics. For the fifteen measurements, the statistical uncertainty of the total flux is  $\delta I/I = 6 \times 10^{-4}$ .

The areal density variation of a 1 cm by 4 cm slab 2 mm thick of  $0.05 \text{ g/cm}^3$  poly(4-methyl-1-pentene) foam (TPX)<sup>10</sup> was measured in five locations across the length of the sample. The results are given in Table I as a function of the distance from one end of the sample, using the first location as the standard. The average variation is  $-2.7 \times 10^{-4} \text{ g/cm}^2$ . This corresponds to a density change of  $0.1 \text{ mg/cm}^3$  if the sample is assumed to have zero thickness variation.

With a  $5 \text{ mCi/mm}^2$  source mounted 6 cm from the detector, three minutes and four hours per  $(0.5 \text{ mm})^2$  area on the sample are required, respectively, to obtain 1% and 0.1% sensitivity to areal density variations in a  $0.05 \text{ g/cm}^2$  plastic sample, 1 cm thick. Total areal density can also

be measured. However, the accuracy of the total areal density measurements is not as good as the measurements of the variations because the x-ray attenuation coefficients as reported in the literature vary by  $\pm 3\%$ . However, more accurate measurements of the total density can be made if one measures the attenuation coefficients more accurately.

Using the  $^{55}\text{Fe}$  x-ray source is safe. There is no chance of a high voltage electrical shock as one would have with a high voltage x-ray generator. This source is stable and unlikely to chip or decompose to spread radioactive particles about the laboratory. It is also chemically non-toxic and will not poison anyone. And the radiation from the source is readily stopped in air or other materials and, therefore, is unlikely to irradiate anyone in the laboratory. The intensity of 6 keV radiation is attenuated by one half in 30 cm of air, 0.06cm of plastic or 20  $\mu\text{m}$  of Al.

The x-ray gauge is easy to use. Measure the transmitted flux at one area on the sample to the accuracy desired. Measure the flux at another area. Compare the two fluxes and calculate the areal density variation.

The x-ray gauge is readily modified to automate the measurements. We plan to mount our samples on a computer controlled x-y stage. The computer will interrogate the scaler-timer. When the timer has shut the scaler off, the computer will read and store the registers in the timer-scaler and move the sample to another position and start another measurement.

The x-ray gauge is portable. Ours is mounted on a 2.5 cm thick by 45 cm by 60 cm aluminum base plate and has one NIM bin of electronics. It can be mounted on a 60 cm by 60 cm rolling cart and moved into any laboratory in our building.

The x-ray gauge measures areal density variations as small as 0.1% in low density plastic foams and other materials. With a spatial resolution as small as 0.5 mm, the system is safe, portable, easy to use and readily lends itself to automated measurements.

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TABLE I

Position (cm)	Counts/sec	Areal Density	66% Confidence
		Variation (g/cm <sup>2</sup> )	Interval (g/cm <sup>2</sup> )
0.5	14.99 ± 0.02	-	
1.0	15.00 ± 0.02	-0.7 × 10 <sup>-4</sup>	1.8 × 10 <sup>-4</sup>
1.5	15.031 ± 0.008	-2.7	0.8
2.0	15.07 ± 0.02	-5.3	1.8
2.5	15.02 ± 0.01	-2.0	1.8

Areal density variations of TPX foam for five locations on 1 cm by 4 cm by 0.2 cm thick sample with 0.05 g/cm<sup>3</sup> average density. Spatial resolution is 0.25 mm<sup>2</sup>. Average areal density variation is -2.7 × 10<sup>-4</sup> g/cm<sup>2</sup>, which could be due to a 1 mg/cm<sup>3</sup> density variation.

## Figure Captions

Fig. 1 X-ray gauge measures attenuation of x-ray flux from a stable source. Differences in x-ray attenuation between different locations in the sample are the result of areal density variations which can be caused by either a dimensional or a density change. The measurement is made with an x-ray beam which is formed by collimating the flux from an  $^{55}\text{Fe}$  source with two collimators. The sample is mounted on a two-axis stage which moves the sample perpendicular to the collimated beam. Photons in the transmitted x-ray beam are detected by a gas proportional counter whose output signal is amplified and routed to a timer-scaler.

## References

1. W. C. Roentgen, Ann Physik u. Chem. 64, 1-37 (1898).
2. E. F. Kaeble, ed., Handbook of X-rays for Diffraction, Emission, Absorption and Microscopy, (McGraw-Hill Book Co., New York, 1967).
3. H. A. Liebhafsky, H. G. Pfeiffer, E. H. Winslow, P. D. Zeman, X-ray Absorption and Emission in Analytical Chemistry (John Wiley & Sons, New York, 1960).
4. H. H. Murray and W. D. Johns, Tappi, 44, 217-219 (1961).
5. T. M. Henderson, D. E. Cielaszyk, and R. J. Simms, Rev. Sci. Instrum. 48, 835-840 (1977).
6. D.M. Stupin, K. R. Moore, G. D. Thomas, and R. L. Whitman, J. Vac. Sci. Technol., 20, 1071-1074 (1982).
7. R. L. Whitman, J. Vac. Sci. Technol., 20, 1359-1361 (1982).
8. X. Clement, A. Coudeville, P. Eyharts, J. P. Perrine and R. Rouillard, J. Vac. Sci. Technol. A, 1, 949-951 (1983).
9. R. H. Day, T. L. Elsberry, R. P. Kruger, D. M. Stupin, R. L. Whitman in Eight International Conference on X-Ray Optics and Microanalysis (Science, Princeton, NJ, 1979), pp 275-282.

10. A. T. Young, D. K. Moreno, R. G. Marsters, J. Vac. Sci. Technol.,  
20, 1094-1097 (1982).

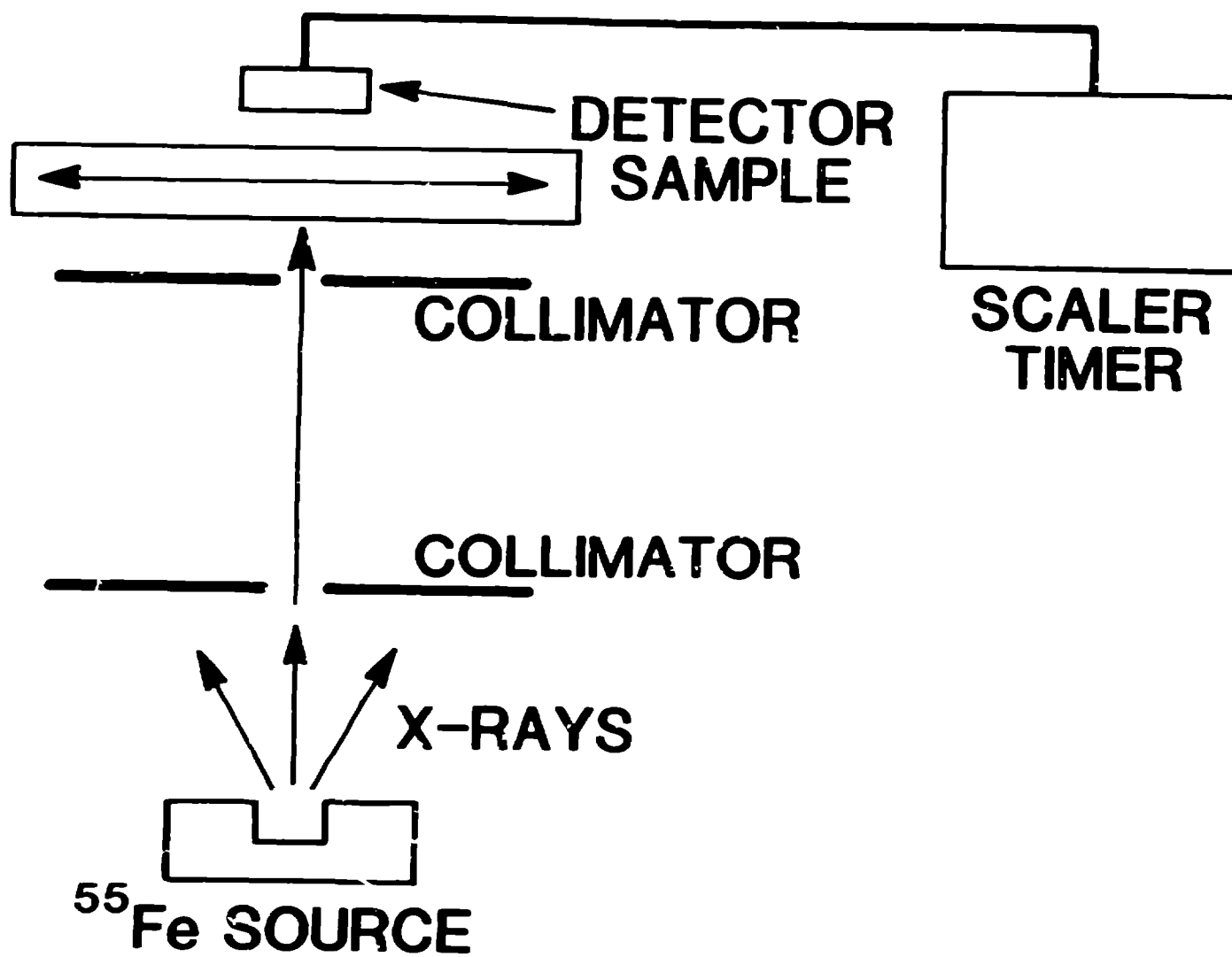


Figure 1