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SUB-KILOVOLT X-RAY CALIBRATION
OF PHOTOGRAPHIC FILM

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SUB-KILOVOLT X-RAY CALIBRATION
OF PHOTOGRAPHIC FILM

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ABSTRACT

The x-ray spectroscopy of laser-generated plasmas can be applied to photon energies of less than 1 keV, provided calibrations of the response of energy discriminators and detectors are performed in the sub-kilovolt region. In our application we use photographic film to monitor the intensity of radiation dispersed by a curved lead stearate crystal. We have determined the response to sub-kilovolt x-rays of two types of film, Kodak No-Screen and the special vacuum ultra-violet Kodak type 101-01. This calibration was obtained by exposing samples of film to continuous beams of nearly monochromatic characteristic radiation from an array of targets. The fluorescent radiations were the K series of carbon and magnesium and the L series of titanium, nickel, cobalt and copper. The response data are presented as a series of characteristic curves, plotting for each film type and photon energy the net diffuse photographic density as a function of the photon flux incident on the film. The processing of all film was in accordance with the manufacturer's suggestions. We attempt to characterize the accuracy and reproducibility of these data. The data demonstrate the greater sensitivity of the type 101-01 to sub-kilovolt x-rays.

The intensity and spectral purity of the incident fluorescent beams were monitored by a thin-window gas proportional counter that used flowing propane gas at sub-atmospheric pressures. We describe the procedures and results of a determination of the quantum efficiency of this counter as a function of photon energy.

INTRODUCTION

The analysis of low-energy x-ray spectra emitted by flash sources has been obtained by Birks and others using a spectrograph incorporating a curved single-crystal disperser and photographic film (1). We are presently developing an instrument that would apply this technique to sub-kilovolt spectra as well, using a lead stearate crystal built up on a flexible substrate. In the course of this project we have experimentally determined the response of two sensitive photographic films to sub-kilovolt x-rays. These films are Kodak No-Screen and the special Kodak type 101-01.*

Our data consist of measurements of the photographic densities produced by a series of exposures of the films to continuous and nearly monochromatic beams of x-rays. Six values of photon energy ranging from the characteristic radiation of carbon at 0.28 keV to that of magnesium at 1.25 keV were used. The principal part of our analysis consisted of correlating the exposure data for each energy to an analytical expression for the characteristic response curve of the films. The degree of success with which these correlations fitted the data is used to assess the accuracy and reproducibility of the photographic process as we applied it. For low- and medium-energy x-rays the photographic density D resulting from an exposure to an x-ray dose E is well fitted by

$$D = D_{\max} [1 - \exp(-\alpha E)],$$

where $D_{\max}$ is the maximum density. This formula is applied under the assumption that it takes only one photon absorbed in an emulsion grain to render that grain developable. Some investigators have suggested, though, that the single photon rule may lapse in the sub-kilovolt x-ray region (2). We initially applied to our data a more general expression developed for exposure of film to visible light and accounting for the contributions to the film darkening of classes of grains of different intrinsic sensitivities (3). We soon found, however, that the analytical form derived from the single-photon assumption was completely satisfactory in fitting our data.

In the theory of optical exposures the parameter α in the exponential is identified with the average area of the emulsion grains. It is thought to represent the total interaction cross-section for activation of the grains, implying that every photon

*Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Atomic Energy Commission to the exclusion of others that may be suitable.

striking a grain participates in the activation, and to be the increment by which the opacity of the film is increased by the development of a grain. In the case of exposures by soft x-rays, however, the value of the parameter will be sensitive to the diminished probability that a photon has of penetrating to and being absorbed in a grain and to the enhancement of the signal at a grain in the emulsion due to electrons knocked out of the supercoat, film base or adjacent grains.

EXPOSURES

The two films studied, Kodak No-Screen and type 101-01, are especially sensitive to low-energy x-rays. The emulsion layers of No-Screen are reported to be about 40 microns thick and to consist of grains having an average diameter between 1 and 1-1/2 microns (4). The emulsions are protected by a gelatin supercoat of slightly less than 1 micron thickness. The special Kodak type 101-01 differs distinctly in construction. It incorporates a single emulsion layer centrifuged onto a gelatin substrate and left unsupercoated. Figure 1 is a scanning electron micrograph of the surface of the film made at the Lawrence Livermore Laboratory (LLL). It shows that the emulsion is a monolayer of grains having an average diameter of about 0.7 microns. The absence of a supercoat enhances the film's response to soft x-rays. However, the film seems to be especially sensitive to variations in the exposure and development procedures. This effect appears in our data as a loss of accuracy and reproducibility for this film compared to No-Screen.

X-Ray Source

Samples of these films were exposed in a vacuum chamber to beams of sub-kilovolt characteristic radiation. The source of the radiation was either one of several interchangeable anodes in an x-ray tube of the Henke design or a fluorescer driven by the Henke tube. Table I lists the series of radiators and their effective

TABLE I

Radiation	Effective photon energy (keV)
magnesium K	1.254
copper L	0.930
nickel L	0.851
cobalt L	0.777
titanium L	0.453
carbon K	0.282

Figure 1. Scanning electron micrograph (at 10,000 $\times$) of the front surface of undeveloped Kodak type 101-01 film.

photon energies. As monitored by a flowing-gas proportional counter, the long-term stability of the source was characterized by a standard deviation of less than 1% in the x-ray flux measurement. The principal spectral contamination observed was due to continuum radiation in the neighborhood of the characteristic peak and, on the average, accounted for 20% of the flux. The typical error in the flux measurement is estimated to be 11%, the chief source of error being the uncertainty in the detector quantum efficiency due to the blending of the continuum with the characteristic radiation.

Counter Calibration

The proportional counter was a Siemens side-window detector fitted with a $75 \text{ } \mu\text{g}/\text{cm}^2$ Formvar window. The counting gas was propane at an absolute pressure of 250 mm. The use of sub-atmospheric gas pressures peaked the counter response in the sub-kilovolt region and reduced the stress on the thin window. The x-ray absorption of the gas was characterized by the following procedure. While the counter was exposed to a constant source of monochromatic radiation, the gas pressure was raised in steps from 50 to 350 mm. At each step the gas amplification was restored by manipulation of the counter high voltage. The variation in the count rate from step to step should thus have resulted only from the increased x-ray absorption in the gas. This assumption was tested by fitting the count rate data to the Beer's-Lambert absorption law, using the values of the absorption coefficients for propane derived by Henke (5). In Figure 2 the data and the curve derived from the fitting analysis are plotted together for several values of photon energy, with the data normalized by an incident count rate determined by the analysis. The agreement is good. From this characterization of the gas absorption and from measurements of the x-ray transmission of the counter window foil we have calculated the quantum efficiency of the proportional counter. The results of this calculation are shown in Figure 3.

Film Processing

After the exposures the films were processed in the manner suggested by the manufacturer. For the No-Screen, development proceeded in RXR at 72° for 4 minutes; for the type 101-01, in D19 at 68° for 4 minutes. After processing, the exposure and background densities were read on a MacBeth singly diffuse transmission densitometer. The sampling aperture of this instrument is an $1/8$ inch diameter circle.

Figure 2. Fraction of x-ray beam absorbed in the propane gas counter over a range of gas pressures, for iron L (0.71 keV), nickel L (0.85 keV), copper L (0.93 keV), and magnesium K (1.25 keV) radiations.

Figure 3. Calculated quantum efficiency of the propane gas counter as a function of photon energy.

RESULTS AND DISCUSSION

The results of the exposure series are shown in Figures 4, 5 and 6, in which the net photographic density is plotted against the incident photon flux for each exposure set. These are the smoothed curves resulting from the analysis applied to the data. The parameters α and $D_{\max}$ of each curve are given in Table II. Also listed is a measure of the success with which each analytical expression predicts the flux required to produce a given density. This measure is the deviation between the flux level monitored in the experiment and that predicted by the analytical formula, averaged over all the experimental data points along the curve. For the No-Screen film the standard deviation over all six values of photon energy is 5.4%, while for type 101-01 film it is 11.1%. From the larger amount of scatter of the data points from the curve seen for type 101-01 we attribute to that film a greater sensitivity to variations in handling exposure, and processing.

While the photon sensitivities of the two films are nearly identical at the highest value of energy (1.25 keV) at which the calibration was performed, the response of the No-Screen falls off much more quickly than the type 101-01 film as the photon energy decreases. For example, the No-Screen sensitivity is diminished by a factor of twenty going from the magnesium calibration curve to the titanium curve at 0.45 keV. The corresponding factor for the type 101-01 film is somewhat less than two. This behavior results in the type 101-01 film being significantly more sensitive in the sub-kilovolt region than the No-Screen. The No-Screen film experiences an increase in sensitivity going from the titanium curve at 0.45 keV to the carbon curve at 0.28 keV. The magnitude of the increase is commensurate with the enhanced transmission of a gelatin supercoat to be expected below the carbon absorption edge. The presence of several silver M edges in this energy region may also have some effect on the sensitivity jump.

TABLE II

No-screen film				Type 101-01 film			
Energy	α	$D_{\max}$	Std dev (%)	α	$D_{\max}$	Std dev (%)	
1.254 keV	9.44×10^{-9}	1.45	± 3.7	1.44×10^{-8}	1.11	± 16.5	
0.930 keV	4.55×10^{-9}	1.48	7.6	1.19×10^{-8}	1.14	10.1	
0.851	3.60×10^{-9}	1.15	5.0	1.04×10^{-8}	1.14	9.2	
0.777 keV	3.57×10^{-9}	0.91	5.1	1.17×10^{-8}	1.08	14.0	
0.453	1.85×10^{-9}	0.43	5.7	8.05×10^{-9}	1.20	7.8	
0.282	$(\alpha \cdot D_{\max} = 1.424 \times 10^{-9})$		5.3	1.63×10^{-8}	0.85	8.8

Figure 4. Response of Kodak No-Screen and type 101-01 films to magnesium K (1.254 keV) and carbon K (0.282 keV) radiations.

Figure 5. Response of Kodak No-Screen and type 101-01 films to copper L (0.930 keV) and cobalt L (0.777 keV) radiation.

Figure 6. Response of Kodak No-Screen and type 101-01 films to nickel L (0.851 keV) and titanium L (0.453 keV) radiations.

It would be expected that as the penetrating power of radiation declined with decreasing photon energy in the sub-kilovolt region, thinner depths near the front surface of a thick emulsion would be effective in the photographic process. This would imply a wastage of radiation in these thin layers and hence an early signal saturation. Indeed, our data show a steady decline in the value of the maximum density $D_{\max}$ for No-Screen film, going from the magnesium radiation at 1.25 keV to the carbon radiation at 0.28 keV, which may be a symptom of this behavior.

Finally, Figure 7 is a scanning electron micrograph of a sample of type 101-01 film exposed to nickel L radiation at 0.85 keV and processed to a net density of 0.1. The clustering of the developed grains suggests that as for higher energy exposures the predominant activation of the emulsion by sub-kilovolt x-rays occurs as a result of the action of the secondary electron shower that radiates from the site of photon absorption.

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Figure 7. Scanning electron micrograph (at 4000x) of an exposed and processed emulsion of Kodak type 101-01 film.