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X-RAY CALIBRATION OF KODAK NO-SCREEN, TYPE AA, AND
TYPE M IN THE 1-4.5 keV REGION

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X-RAY CALIBRATION OF KODAK NO-SCREEN, TYPE AA, AND TYPE M IN THE 1-4.5 keV REGION

The response of three Kodak x-ray films, No-screen, type AA and type M has been measured at .93, 1.5 and 4.5 keV. These measurements were motivated by a need for a radiographic calibration applicable to the analysis of x-ray images produced by an x-ray microscope.¹ This instrument is in current use to study x-rays generated by laser-induced plasmas. By appropriate filter selection, the microscope will focus narrow energy bands between .5 and 8 keV.

TECHNIQUE

The experimental arrangement shown in Figure 1 consisted of a Henke type x-ray tube, film chamber and proportional counter. The x-ray tube section was vacuum coupled to the beam pipe by a thin Formvar window ($\sim 55 \mu\text{gm}/\text{cm}^2$) allowing the tube to operate at 10^{-6} Torr. A vacuum of less than 10 microns was maintained throughout the rest of the system during the exposures.

The Henke tube generates x-rays by electrostatically focusing electrons from a tungsten filament hidden behind the anode to a small area on the front surface of the anode. This geometry protects the anode target area from tungsten contamination by the filament. Interchangeable anodes permit selection of a variety of targets. For this calibration, Copper, Aluminum and Titanium anodes were used to produce $\text{Cu}(\text{L}\alpha)$, $\text{Al}(\text{K}\alpha)$ and $\text{Ti}(\text{K}\alpha)$ lines. Bremsstrahlung background extending in energy to the

anode potential was essentially eliminated by thin filters of the same material as the anode positioned 15" forward of the film plane. Filter thickness and anode voltage were adjusted to maximize spectral purity. A typical spectrum for an aluminum target showing discriminator settings is shown in Figure 2. Similar spectral shapes were obtained for copper and titanium anodes.

The film was exposed to a collimated beam 1.4 cm x 1.4 cm. Exposures varied from 1 minute to a maximum of 270 minutes. Periodic monitoring of the longer exposures was accomplished by a fixture enabling the film holder to be moved in and out of the beam within the vacuum chamber. The fixture permitted accurate replacement of the film to continue the exposure after sampling.

Exposures were monitored by a proportional counter 37 cm x 7 cm x 5.1 cm in depth located in a separate vacuum unit attached to the end of the beam pipe. A continuous flow of P-10 gas flowing through the counter was held to a constant pressure of 1.2 atmosphere. X-rays entered the detector through a small aperture to keep the count rate at a reasonable level. They then passed through a 60 $\mu\text{gm}/\text{cm}^2$ Formvar window supported by Buckbee-Mears 70% transmission mesh. The detector was initially calibrated by a set of alpha-excited fluorescers producing lines from .28 to 4.5 keV. A miniature aluminum anode x-ray tube mounted in the counter was used to periodically check the calibration. Table I gives the filter thickness and counter window area used for each target.

TABLE I

Anode	hv(keV)	Filter	Aperture Area(cm ²)	Window Transmission		Calculated Efficiency
				Calculated	Measured	
Cu	0.934	Cu(1.3x10 ⁻⁴ cm)	9.25x10 ⁻⁴	0.83	0.80	1.00
Al	1.487	Al(1.0x10 ⁻³ cm)	3.97x10 ⁻³	0.93	0.93	1.00
Ti	4.50	Ti(3.8x10 ⁻³ cm)	6.47x10 ⁻²	1.00	-	0.988
Ti	4.50	Ti(3.8x10 ⁻³ cm)	6.57x10 ⁻³	1.00	-	0.988

A supplemental calibration using an x-ray radiation source was performed to obtain additional points for No-Screen film at 5.9 keV. A 10 millicurie Fe⁵⁵ source mounted in a holder with fixed collimation was monitored by the proportional counter and then used to expose the film. The source geometry for both film and proportional counter was identical.

All films were processed in Kodak Rapid X-ray (RXR) developer for 6 minutes at 72°F.

Net specular densities were obtained by scanning each exposure with a Photometric Data Systems, Series 1000, microdensitometer using a ten power objective having a numerical aperture of 0.25. The scan slit was 10 microns by 10 microns. This unit stores the digitized results on magnetic tape for further analysis and is used to scan the micrographs of laser induced plasmas produced at Lawrence Livermore Laboratory.

Since the Series 1000 microdensitometer measures specular density, whereas published results are often given in diffuse density, both spectral

and diffuse data were taken for each calibration image. A Macbeth TD-504 macro-diffuse densitometer using a Wratten 106 filter was used to measure the net center-spot diffuse density.

Values for specular density versus diffuse density are plotted in Figure 3 for No-Screen film. A second order polynomial has been fit to the data and is plotted as a solid curve. To allow the conversion of density units for film types AA and M, the coefficients for the second order polynomials which give the best fits to the data are listed in the insert in Figure 3. It should be cautioned that specular densitometers may have different acceptance angles of transmitted light (differing numerical apertures), yielding different densities for the same photographic exposure.

EXPERIMENTAL DATA

The calibration results are shown in Figures 4 through 6. Values for the net specular density have been plotted against the exposure in ergs/cm^2 for the three film types at 0.93 keV, 1.49 keV and 4.5 keV. In addition, data for No-Screen at 5.9 keV is given in Figure 7.

All data for the three film types were taken at one energy. The anode was then changed and data for all films were acquired at the new energy. Reproducibility checks made by repeating exposures several days or weeks following an initial exposure show agreement to within $\pm 5\%$.

COMPARISON WITH OTHER RESULTS

Direct comparison of these data with other results is difficult. Slight differences in processing techniques (age of developer, hand versus mechanical agitation, temperature control, etc.) can change the density for a given exposure by a factor of two. Comparing results obtained with different

types of densitometers can also lead to apparent variations in excess of 50%. The comparison of data sets corrected for processing differences is being examined by R. A. Armistead at the Stanford Research Institute. Numerous calibrations have been made for No-Screen film. Armistead's preliminary results¹⁰ show good absolute agreement (20%) between the data in Figure 6 and other experimenter's measurements at similar energies.

We now compare the energy dependent variation in the characteristic curve's shape for No-Screen film to other results. No comparisons will be made for film types AA or M since the energy interval 0.9 to 4.5 keV, no other measurements have been found.

The No-Screen data in Figures 4 through 6 is replotted in Figure 8. Unlike measurements above 6 keV, these data show the shape of the characteristic curve varies with energy between 1.5 keV and 4.5 keV. This shape change is also seen by comparing characteristic curves from other measurements. The solid curves in Figure 8 are normalized data from three other investigations taken at or near the energies used in this calibration. These data all show similar variation in shape in this energy interval.

FILM COMPOSITION

To calculate the x-ray interaction with the film, the composition of the films was determined with several techniques.

A schematic representation of the film is shown in Table II. All films used in this investigation are double sided, consisting of a layer of supercoating, emulsion, substraat, emulsion and supercoating. The supercoating thickness and composition were supplied by Kodak.⁹ The thickness of Emulsion and Estar substraat were determined by weighing a known

area of film before and after removing the emulsion with a weak sodium hydroxide solution. The weight fractions of Carbon, Hydrogen and Nitrogen for the emulsion were determined with a cryogenic vacuum analyzer. The amounts of silver and bromine in the emulsion were determined by measuring the x-ray transmission at energies just above and below the K-absorption edges for silver and bromine. An independent measurement of the silver content in the emulsion was determined by controlled potential coulometry. All of the remaining material in the emulsion was assumed to be oxygen. Typical uncertainties on the measured quantities are: Thickness, $\pm 10\%$; Carbon, Hydrogen, Nitrogen and Silver, $\pm 1\%$; Bromine, $\pm 15\%$. The measured film compositions are summarized in Table II.

TABLE II

	↓
Supercoating	A
	↑
Emulsion	B
	↓
	↑
Estar	C
	↓
	↑
Emulsion	B
	↓
Supercoating	A
	↑

FILM TYPE

NO-SCREEN		AA	M
Thickness: (mg/cm ²)			
A	0.1	0.1	0.1
B	6.4	3.4	3.2
C	24.4	24.6	24.8
Emulsion Composition (mass fraction)			
C	.214	.206	.202
H	.038	.029	.029
O	.186	.154	.162
N	.057	.069	.070
Ag	.290	.312	.307
Br	.22	.23	.23

MASS FRACTION

	C	H	O	N
SUPERCOATING	.433	.077	.0375	.155
ESTAR	.625	.042	.333	-

ENERGY SENSITIVITY

The relative sensitivity of a film can be expressed as the reciprocal of the exposure (photons/cm²) necessary to produce a constant density. A sensitivity curve for No-Screen film obtained from measurements made at this laboratory is shown in Figure 9. Density increases linearly with exposure at low exposures. The deviation from linearity occurs when the exposure reaches a level where photons are absorbed in grains that already have been rendered developable. The density characterizing this transition from linearity increases with increasing x-ray penetration. This density at 1 keV and 10 keV is ~0.4 and ~2.0, respectively. In order to keep in the linear region for all the data in Figure 9, a density of 0.1 was chosen.

Plotted as a dashed curve in Figure 9 is the relative probability that a photon will interact in the emulsion.

The low energy data show greater absorption than would be expected from the 0.1 mg/cm² supercoat thickness specified by KODAK. Since the chemical composition of the supercoat does not differ greatly from the emulsion base, we have used 0.4 mg/cm² as the effective thickness of supercoat in the calculations.

The enhanced sensitivity above the single interaction curve for energies greater than 10 keV is due to the increase in electron range. The electron range varies from 0.1 mg/cm² at 5 keV to 20 mg/cm² at 100 keV. Since the nominal separation between grains in x-ray films is about 2 mg/cm², the electrons in this energy interval have sufficient range to interact with several grains.

The absorption coefficients from Henke⁷ and McMasters⁸ have been used in calculating the x-ray interactions in the film. Henke's data has been used for energies less than 1.5 keV and McMaster's data above 1.5 keV.

A MODEL FOR CALCULATING FILM SENSITIVITY

The calibration curves in Figures 4 through 7 can be used directly for exposures at 0.9 keV, 1.5 keV and 4.5 keV. The extrapolation to other energies and/or densities requires the use of a suitable model. If the energy dependence of the model parameters is understood, the model can be used to extrapolate the calibration to energies outside the range of measured data with confidence. Since the calibration data was measured only at three energies, a model parameterizing all the energy dependent photographic processes is not justified. We have chosen a simple three parameter model to fit the data at each energy. The parameters are maximum density, film sensitivity and the fraction of developed grains that required more than one photon interaction.

Three assumptions have gone into the model: 1) The interactions with the grains of silver bromide follow Poisson statistics. 2) Only the energy deposited in the silver bromide contributes to the density. 3) After a grain is rendered developable, subsequent deposition of energy in that grain will not change the density. Arguments both for and against these assumptions can be found in the literature.⁶

These assumptions lead to the following expression relating density to exposure.

$$D = A(1)[1 - \exp(-A(2)E) \left\{ 1 + A(2)E + (A(2)E)^2 + \dots + \frac{(A(2)E)^{n-1}}{n!} \right\}]$$

Where

D = density/cm²

$A(1)$ = maximum density

$A(2)$ = film sensitivity

n = number of interactions necessary to render
a grain developable

E = exposure in ergs/cm²

Since almost all x-ray interactions with silver bromide should render the grain developable (n should be no greater than 2), the function fit to the data was:

$$D = A(1) (1 - \exp(-A(2)E) [1 - A(3)A(2)E])$$

where: $A(3)$ represents the fraction of events requiring 2 interactions.

The parameters $A(1)$, $A(2)$ and $A(3)$ were derived from the data by using nonlinear least square fitting techniques. The model is compared with the measured data in Figures 4 through 6, plotted as solid lines. In general, the comparison is good.

The best-fit parameters for the No-Screen film model are shown in Figure 10. $A(2)$ was constrained to be equal to the calculated film sensitivity indicated by the solid curve in Figure 9, while $A(1)$ and $A(3)$ were allowed to vary. The energy variation of $A(1)$ is seen approximately $E^{1/2}$. A similar energy dependence on the maximum density has been observed for electron exposures.⁶

Since the emulsion is essentially black between 3 keV and 4 keV, the structure in the parameters due to the L-absorption edges of bromine should be small. The interpretation of $A(3)$ as the fraction of 2 hit events is seen to be incorrect since it becomes negative for energies less than 1.5 keV.

The parameters obtained from fitting the data for film types AA and M illustrate an additional short-coming of the model. $A(3)$ in all cases is essentially zero, reducing the model to a two parameter model.

The product of $A(1)$ and $A(2)$ is equal to the slope of the calibration curve in the linear (low density) region. This slope is accurately determined by the data. If one tries to equate $A(2)$ to the film sensitivity, $A(1)$ (the maximum density) is forced to be unacceptably too small.

Although the lack of a physical interpretation of the parameters places extrapolation to higher or lower energies on less firm ground, the value of the model is seen in Figures 4 through 7 in providing good fits to the experimental data. Since the structure in the parameters, due to its absorption edges in bromine is expected to be small, the model can be used to calculate calibration curves in a limited energy interval. The energy dependence of the parameters for film types AA and M are shown in Figures 11 and 12. The curves in these figures are seen to be quite different than those in Figure 10.

Only three exposures were measured for film type M at 4.5 keV. These three points are in the linear region of the response curve and do not allow sufficient accuracy in determining the model parameters. The unrealistically high value of 31.0 for parameter $A(1)$ changes significantly with a 20% change in any of the three data points. In order to interpolate between 1.5 and 4.5 keV, more measurements at higher densities at 4.5 keV are necessary.

CONCLUSIONS

The Kodak x-ray films No-Screen, AA and M have been calibrated at

three energies over a range in densities. These data have been fit with an analytic model which extends the calibration to densities and energies other than those measured. The estimated uncertainties on the measured values are typically $\pm 15\%$. Using the model at the measured energies to interpolate exposures for densities within the range measured is also typically good to $\pm 15\%$. When extrapolating to densities greater than those measured the uncertainties may become much greater due to saturation effects. Calculation of calibration curves for energies other than those measured, with the use of Figures 10 through 12 can be done with less certainty. The energy interval and density interval which the model can be used to calculate exposures is given in Table III. The uncertainty in the calculated exposure in this range is less than a factor of two.

TABLE III

<u>Film Type</u>	<u>Energy Interval</u>	<u>Density Interval</u>
No-Screen	0.7-7.0	0 - 3.0
AA	0.9-6.0	0 - 4.0
M	0.9-4.5	0 - 2.5

FOOTNOTES

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- 10) R. Armistead, SRI 5-4227 document number.
- 11) Bonn and Slivinsky, Internal Report, VWS 69-1 (1969).

Figure 1

Figure 2

Figure 3

Figure 4.

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Figure 12

- Figure 1 Calibration geometry showing positions of x-ray source, film and proportional counter.
- Figure 2 Typical pulse height spectrum illustrating spectral purity. Shown here are the spectrum from the aluminum anode and the discriminator settings defining the scaler window.
- Figure 3 Relationship between specular and diffuse density for No-Screen film. Coefficients for polynomial fit for the three film types are shown in insert.
- Figure 4 Characteristic curves for No-Screen, AA and M films at 0.93 keV. Measured data are indicated by symbols and solid lines are calculated from the model.
- Figure 5 Characteristic curves for No-Screen, AA and M films at 1.487 keV. Measured data are indicated by symbols and solid lines are calculated from the model.
- Figure 6 Characteristic curves for No-Screen, AA and M films at 4.5 keV. Measured data are indicated by symbols and solid lines are calculated from the model.
- Figure 7 Characteristic curve for No-Screen film at 5.9 keV. Measured data are shown by points. Solid curve is calculated from model.
- Figure 8 Characteristic curves for No-Screen film showing shape variation with energy. Measured data from this calibration indicated by: Triangles, = 0.93 keV; crosses, = 1.5 keV; circles, = 4.5 keV; These data are compared with normalized data from other investigations as indicated in the legend.
- Figure 9 Relative sensitivity curve for No-Screen films obtained from LLL data. Measured data are plotted as points. Solid curve is the relative single interaction probability in the emulsion calculated from the film composition.
- Figure 10 Energy dependence of model parameters for No-Screen film. $A(2)$ equals the calculated film sensitivity.

Figure 11

Model parameters for AA film. The data is well fit with two parameters.

Figure 12

Energy dependence of model parameters for type M film. Two parameters are sufficient to fit the data.

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