LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

LLNL-JRNL-451153

Calibration of a High Resolution Grating Soft X-ray Spectrometer

E. W. Magee, J. Dunn, G. V. Brown, K. V. Cone, J. Park, F. S. Porter, C. A. Kilbourne, R. L. Kelley, P. Beiersdorfer

August 25, 2010

Review of Scientific Instruments

Disclaimer

Calibration of a High Resolution Grating Soft X-ray Spectrometer [a)]

E. W. Magee,[1] J. Dunn,[1,b)] G. V. Brown,[1] K. V. Cone,[1, 2] J. Park,[1, 2] F. S. Porter,[3] C. A. Kilbourne,[3] R. L. Kelley,[3] and P. Beiersdorfer[1]

[1]*Lawrence Livermore National Laboratory, P.O. Box 808, Livermore, California 94550, USA*
[2]*Department of Applied Sciences, University of California, Davis, California 95616, USA*
[3]*NASA/Goddard Space Flight Center Greenbelt, MD 20770, USA*

(Presented XXXXX; received XXXXX; accepted XXXXX; published online XXXXX)
The calibration of the soft x-ray spectral response of a large radius of curvature, high resolution grating spectrometer (HRGS) with a back-illuminated charge-coupled device detector is reported. The instrument is cross-calibrated for the 10 – 50 Å waveband at the Lawrence Livermore National Laboratory electron beam ion trap (EBIT) x-ray source with the EBIT calorimeter spectrometer (ECS). The HRGS instrument is designed for laser-produced plasma experiments and is important for making high dynamic range measurements of line intensities, line shapes and x-ray sources.

I. INTRODUCTION

The development of x-ray spectroscopic instrumentation continues to be important for further understanding of the processes and physics in high temperature and high energy density plasmas. The measurement and correct interpretation of soft x-ray spectra, relative line intensity ratios, continuum slopes, spectral line shapes and source x-ray emissivity, can yield a wealth of information on the plasmas.[1–4] This invariably requires a methodology for calibrating the instrument sensitivity function over the waveband of interest. For soft x-rays in the 10 – 500 Å range, grating spectrometers, for example,[5] are frequently fielded. The calibration can be technically challenging as instruments using grazing incidence mirrors or gratings are susceptible to the contamination of thin hydrocarbon layers where even 10 nanometer coatings can have a significant effect on the reflectivity. Also the use of thin sub-micron filters, which may be affected by oxidation or have non-uniform thickness, for light tightness or signal attenuation require the transmission to be accurately determined.[6] The detection efficiency of charge-coupled device (CCD) detectors using back-thinned chips can be affected by thin dead layers of silicon and silicon oxide.[7]

We report the calibration of a High-Resolution variable-spaced Grating, flat-field Spectrometer (HRGS) [8,9] with a back-thinned CCD detector for the wavelength range of 10 – 50 Å on the electron beam ion trap (EBIT) x-ray source at the Fusion and Astrophysics (FAST) data and instrument calibration facility at the Lawrence Livermore National Laboratory (LLNL).[10,11] The EBIT calorimeter spectrometer (ECS) [12] was used to cross-calibrate the grating instrument on EBIT-I by recording the soft x-ray emission simultaneously. We show an example of the recorded spectra from both instruments. The HRGS efficiency, ε, defined here as *grating reflectivity* × *quantum detection efficiency* product, is measured to be 1 – 1.6 % for the 15 – 30 Å wavelength range by taking into account the solid angles, source emission volume and filters used in the calibration run for the two instruments. We describe the methodology involved in the calibration, some of the limitations as well as areas that will be pursued in the future.

II. EXPERIMENTAL DESCRIPTION

The HRGS soft x-ray spectrometer consists of a 2400 line/mm variable spaced grating of dimensions 5 × 10 cm^2 with a radius of curvature of R=44.3 m to disperse the incident spectrum in a flat-field detection plane.[8] The HRGS spectrometer has recorded spectra on EBIT-I and laser-produced plasma x-ray sources.[8,9] The calibration of the HRGS instrument was performed at FAST using EBIT-I. Various source gases including CO_2, N_2, and Ne were injected into the trap at a nominal pressure of 10^{-6} Torr. These generated K-shell soft x-ray emission lines of interest in the 10 – 50 Å wavelength range mostly from the H-like and He-like charge states. The continuous electron beam, nominally of 60 μm beam diameter, was held at a constant energy and ionized and excited the gas in the trap to the K-shell ion stage. The ion trap region and x-ray emission region was nominally 2 cm in length and defined axially by the voltages applied to the electrodes in the drift section. The ion trap voltage was pulsed to periodically empty the trap and duration time was defined by the cycle time. The electron beam energy and current together with the cycle time were optimized to maximize the x-ray source emission: 3.08 keV, 132 mA and 49 ms was used for the CO_2 and N_2 gases while the Ne gas was run at 5.0 keV, 76 mA and 105 ms.

The grating spectrometer and the ECS, separated by 90°, viewed the x-ray source in the horizontal plane. The grating spectrometer was run without a slit to maximize the x-ray signal at the detector and was orientated so that the source was parallel to the groove direction. The source size and x-ray emission region therefore determined the instrument spectral resolution. A series of baffles between the source and the grating were set to illuminate the grating and reduce scatter inside the spectrometer. No light tight filter was required. The x-ray source to grating center was measured to be 194.6 cm. The spectrometer grating to detector distance was adjusted to achieve the best focus of the spectral lines and was approximately 150 cm depending on the wavelength setting. The grating surface was inclined at an angle of 2° to the source. A Princeton Instruments, liquid-Nitrogen-cooled (operated at -110°C) back-thinned CCD detector was placed at the flat-field detection plane. The CCD contained a

[a)]Contributed paper published as part of the Proceedings of the 18th Topical Conference on High-Temperature Plasma Diagnostics, Wildwood, New Jersey, May, 2010.
[b)]Electonic mail: dunn6@llnl.gov.

1340 × 1300 pixel array with each pixel having a 20 × 20 μm^2 dimension. The x-ray calibration runs typically lasted 60 minutes per exposure to increase the total number of detected x-ray photons. The CCD signal was digitized by a low noise 16-bit 50 kHz analog to digital convertor (ADC) at the high gain setting, *g*, of 1.07 elec./ADC count. The recorded 2-dimensional CCD images were analyzed as follows: A background image was numerically subtracted and a small tilt angle of 0.15° was applied to the spectral lines to align with the CCD array. Cosmic ray events were removed in 2 stages by pixel value analysis threshold techniques to set hot pixels to background values followed by nearest pixel value comparisons. This was found to effectively remove most of the cosmic rays with a very small effect of reducing the peak x-ray emission lines by less than 2%. The signal was then averaged over the full height of the line (1290 pixels) to generate a spectrum. The mainly H-like and He-like ion emission lines were identified and a wavelength dispersion applied using the reference values tabulated in Kelly.[13] The measured line signal, *S*, in ADC counts was converted to detected photons, *N*, using the relation $N = (S\ g\ \omega)/E$ where g is the above ADC gain, ω is the Si work function of 3.65 eV/elec. and *E* is the photon energy.

The EBIT calorimeter spectrometer is a solid-state energy dispersive device first developed at NASA's Goddard Space Flight Center in 1984.[12] The present ECS consists of a 6 × 6 array of HgTe pixels cryogenically-cooled to 50 mK using an adiabatic de-magnetization refrigerator in a liquid $^3He/^4He$ bath. The array consists of 624 × 624 μm^2 × 8 µm thick pixels for mid-energy 0.1 – 10 keV photons interspersed with 624 × 500 μm^2 × 100 µm thick pixels for improved high energy 0.5 – 100 keV photon detection. HgTe is chosen as the detector material because of high x-ray absorption and low heat capacity. X-ray absorption is 100% for photon energy below 4 keV. The energy resolution when operated at the 50 mK cryogenic temperature is $\Delta E \sim 5$ eV at 6 keV and $\Delta E \sim 25$ eV at 60 keV photon energy. Thermal isolation of the calorimeter is achieved with 4 thin foils of aluminized polyimide ($C_{22}H_{10}N_2O_5$) with a total thickness of 147.0 nm Al/238.6 nm polyimide. The absorption of this filter set has to be corrected in the calibration for low energy photons under study here. A fifth filter of 21 nm Al/1030nm polyimide was added to attenuate the x-ray signal further onto the ECS to minimize signal pile-up. In the data analysis the signal from 16 of the mid-energy pixels was combined to give high photon statistics. The array was positioned 95.17 cm from the source and observed the center 1.65 cm length of the cylindrical ion beam.

III. RESULTS

A sub-set of the data is reported in this paper to illustrate the cross-calibration technique. The HRGS results have been converted from wavelength to energy scale to directly compare the spectra from the two instruments as shown in Fig. 1 for nitrogen gas ionized by the electron beam. The spectra from both instruments are plotted as total detected photons versus energy. The data were recorded for 60 minutes. The energy range of 400 – 600 eV represents the full spectral coverage of the grating instrument at one setting limited by the CCD detector size. The strong n = 2 – 1 and 3 – 1 lines are labeled as well as some of the higher order He-like ion series. It can be seen that the HRGS spectrum is well resolved. Spectral resolution of E/ΔE~1300 is determined by the EBIT source size and corresponds to ΔE of 0.34eV (FWHM) or equivalent to 3 CCD pixels for the N He-α line at 430.7 eV. Residual oxygen ions are present in the ion trap and this accounts for the weak He-α line at 574 eV. The ECS covers a much larger range (not shown in the figure) and has the advantage of recording photon energies from 100 eV to 100 keV simultaneously. The ECS spectrum (in red) does not resolve the He-β line on the low energy side of the stronger Ly-α line at 500 eV due to the lower energy resolution of ΔE ~5 eV. It can also be noted that the line intensity ratios for the two instruments change across the spectra. The ECS results as shown are not corrected for the filter response and this strongly attenuates the lower photon energies.

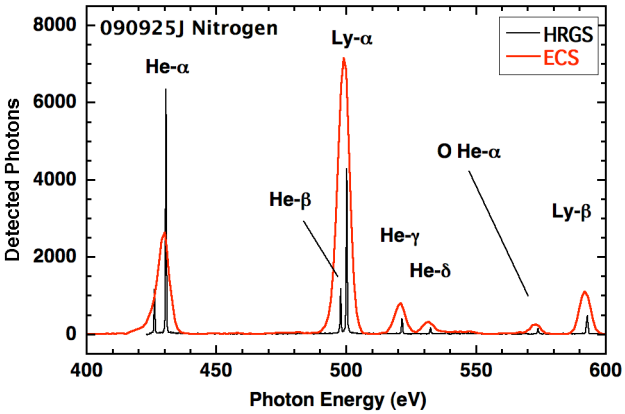

FIG. 1. Soft x-ray nitrogen spectrum recorded from EBIT source showing strong He-like and H-like lines showing detected photons versus photon energy. The ECS spectrum (in red) is shown while the grating spectrum is in black. (Color online)

The strongest x-ray lines for nitrogen and oxygen were analyzed for the two instruments to calibrate the HRGS efficiency, ε, defined here as *grating reflectivity* × *quantum detection efficiency* product for 15 – 30 Å. For this study it was not possible to separate the grating reflectivity from the CCD quantum detection response. The geometry of each spectrometer was taken into consideration including the solid angle subtended to the source, the emission volume observed by the detectors as well as any filter corrections. The ECS solid angle was 6.9 × 10^{-6} steradians. The HRGS instrument solid angle was determined by the effective grating height tilted towards the source in the plane of dispersion (1.79 mrad) and the angle subtended by the detector orthogonal to the plane of dispersion (7.62 mrad). This latter number was measured separately in a laser plasma experiment by placing an aperture in front of the grating close to the x-ray point source to determine (a) the fraction of the grating illuminated by the x-rays reaching the detection plane and (b) the degree of focusing along the spectral line height. It was found that there was no focusing of x-rays in the plane orthogonal to the dispersion. The HRGS solid angle was 1.37 × 10^{-5} steradians or twice the ECS instrument for the EBIT calibration run. Very small corrections (<5%) were made for the observed emission volumes of the two instruments. For some spectral lines the ECS is unable to resolve close lines, as shown in Fig. 1, in which case the line intensity for the grating spectrometer is integrated over the full width observed by the ECS instrument.

Figure 2 shows the grating efficiency as a function of the x-ray wavelength in the 15 – 30 Å range. Values of 1 – 1.6% are measured where the absolute response is gradually decreasing at shorter wavelengths as a result of the falling grating efficiency. The error bars of ~±15% are largely determined by a number of factors but are not limited by the photon statistics in individual

lines. X-ray lines recorded in the grating spectrometer are determined to have wings due to the grating response function from surface roughness effects and are readily observed in the stronger lines. Precise fitting of the line shape is required. Secondly, background photons or low level noise that extend away from the lines that are not wings or part of the line (and may not be in the ECS data) are included in the signal as a result of the wider energy range of the ECS. This becomes important for weaker lines recorded in the calibration. These effects have to be addressed carefully and will require further detailed analysis. Studying high-resolution ECS events could help to determine the overall contribution in the latter case. The third contribution comes from the transmission response of the thin filter set used in front of the ECS. The filter transmission will be calibrated in the near future. It can be noted here that previous filter measurements conducted on the ECS have shown that the filter samples and thicknesses supplied by Luxél have been very precise.[6,14]

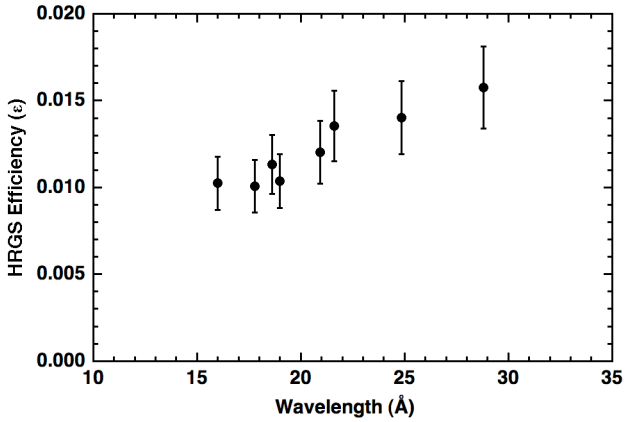

FIG. 2. The HRGS absolute efficiency, ε, defined here as *grating reflectivity* × *quantum detection efficiency* product as a function of wavelength for 15 – 30 Å. ε is found to be ~1 – 1.6%. The grating reflectivity is expected to be very close to the above values.

In previous work to characterize the response of a back-thinned CCD detector, typical values of dead layers of 10 nm of SiO_2 as well as 20 nm Si were determined.[7] The active depth of the detector was also determined to be 10 μm. This would give a predicted detection efficiency that would gradually slope down from 97% at 15Å to about 90% at 30 Å. We can speculate that the values for the grating reflectivity are very close to the efficiency curve in Fig. 2 and so the reflectivity is ~1 – 2%. The future plan is to separately determine the CCD response as well as extend the calibration to a wider spectral range.

IV. CONCLUSIONS

The calibration of a high resolution grating spectrometer has been reported using the FAST facility at LLNL and the EBIT calorimeter spectrometer. This allows the absolute calibration of the main components of the spectrometer namely the grating reflectivity and detector quantum efficiency where values of 1 – 1.6% have been measured. The large area of the grating, allowing increased solid angles, together with the high spectral resolution capability make this a powerful instrument for high temperature plasmas.

V. ACKNOWLEDGMENTS

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

[1]*Plasma Diagnostic Techniques*, ed.s R. H. Huddlestone and S. L. Leonard, (Academic Press, New York, 1965).
[2]H. R. Griem, *Plasma Spectroscopy*, (McGraw-Hill, New York, 1964).
[3]C. De Michelis and M. Mattioli, Nucl. Fus. **21**(6), 677 (1981).
[4]J. D. Kilkenny, R. W. Lee, M. H. Key, and J. G. Lunney, Phys. Rev. A. **22**(6), 2746 (1980).
[5]T. Kita, T. Harada, N. Nakano, and H. Kuroda, Appl. Opt. **22**, 512 (1983).
[6]G. V. Brown, P. Beiersdorfer, J. Emig, M. Frankel, M. F. Gu, R. F. Heeter, E. Magee, D. B. Thorn, K. Widmann, R. L. Kelley, C. A. Kilbourne, and F. S. Porter, Rev. Sci. Instrum. **79**, 10E309 (2008).
[7]Yuelin Li, G. D. Tsakiris, and R. Sigel, Rev. Sci. Instrum. **66**(1), 80 (1995).
[8]P. Beiersdorfer, E. W. Magee, E. Träbert, H. Chen, J. K. Lepson, M. -F. Gu, and M. Schmidt, Rev. Sci. Instrum. **75**, 3723 (2004).
[9]J. Dunn, E. W. Magee, R. Shepherd, H. Chen, S. B. Hansen, S. J. Moon, G. V. Brown, M. -F. Gu, P. Beiersdorfer, and M. A. Purvis, Rev. Sci. Instrum. **79**, 10E314 (2008).
[10]P. Beiersdorfer, Can. J. Phys. **86**, 1 - 10 (2008).
[11]G. V. Brown, P. Beiersdorfer, J. Dunn, E. Magee, J. Park, F. S. Porter, M. Schneider, E. Träbert, R. L. Kelley, and C. A. Kilbourne, "The Fusion and Astrophysics Data and Diagnostics Calibration Facility", in print in the Proceedings of the SPIE Astronomical Telescopes and Instrumentation 2010 Conference (2010).
[12]F. S. Porter, B. R. Beck, P. Beiersdorfer, K. R. Boyce, G. V. Brown, H. Chen, J. Gygax, S. M. Kahn, R. L. Kelley, C. A. Kilbourne, E. Magee, and D. B. Thorn, *Can. J. Phys.* **86**, 231 (2008).
[13]R. L. Kelly, J. Phys. Chem. Ref. Data **16**, 1 (1987).
[14]J. Dunn, P. Beiersdorfer, G.V. Brown and E.W. Magee, in *X-ray Lasers 2008*, Proceedings of the 11th International Conference on X-ray Lasers, 17 – 22 August 2008, Belfast, UK, Springer Proceedings in Physics Vol. **130**, Ed.s C.L.S. Lewis, D. Riley, 461 - 7 (2009).