X-ray and EUV spectral instruments for plasma source characterization

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ABSTRACT

A set of spectral analytic instruments has been developed for absolute intensity measurements in a spectral range of 1-600 Å: 1) several modifications of grazing incidence spectrographs; 2) EUV monochromator- spectrometer with a constant angle of deviation; 3) focusing crystal von Hamos spectrometer using cylindrical mica and pyrolytic graphite crystals and a CCD linear array as a detector. These instruments are useful for plasma diagnostics, x-ray and EUV spectroscopy of laser-generated plasmas and capillary discharge plasmas, x-ray and EUV reflectometry, radiometry and x-ray fluorescence application.

Keywords: x-ray and EUV spectroscopy, laser-generated plasmas, capillary discharge plasmas, bent crystals, non-destructive testing (XRF).

1. INTRODUCTION

Soft x-ray and Extreme Ultraviolet (EUV) spectral ranges are important for fundamental research such as spectroscopy of highly charged ions and diagnostics of high-temperature plasmas. In the past decade extraordinary progress was achieved in soft x-ray and EUV technologies which can be used in the various practical applications—EUV and soft x-ray lithography and microscopy, x-ray fluorescence analysis, EXAFS spectroscopy, etc. All of these uses require effective and versatile spectral devices for source characterization and intensity measurements. In this article, we describe a set of spectral analytic instruments which have been developed for absolute intensity measurements in a spectral range of 1-600 Å. These instruments include several versions of grazing incidence and a focusing crystal spectrometer. The devices are useful both for fundamental research and for practical applications.

2. GRAZING INCIDENCE SPECTROGRAPHS (GIS)

Off-Rowland geometry is used in the spectrographs—the spectra are recorded in a plane perpendicular to the diffracted rays (see Fig. 1). In this method, the exact focusing takes place at only one wavelength λ_0, which corresponds to the intersection point of the registration plane with the Rowland circle. However due to the small numerical aperture of the spectrograph, it is possible to record the spectrum in a wide spectral range $\lambda_0 \pm \Delta\lambda$. The value $\Delta\lambda$ is connected with the observed spectral resolution $\lambda/\delta\lambda$–the wider $\Delta\lambda$ the lower $\lambda/\delta\lambda$ because of defocusing. The alignment to different wavelengths λ_0 is produced by changing the distance between the plane of registration and the grating.

The advantages of this geometry include a simplicity of a design, absence of complicated alignment of optical elements on the Rowland circle and high luminosity at the central wavelength λ_0. The limitations are a narrow enough spectral range of registration $\lambda_0 \pm \Delta\lambda$ limited by the reduction of the spectral resolution due to defocusing.

1-meter spherical replaceable diffraction gratings installed at a 4° grazing angle are used in the spectrographs–1200, 600, 300 groves/mm, all W-coated. The spectral range covered with the use of three gratings is λ=20 – 800 Å. Spectral resolving power $\lambda/\delta\lambda$ at the central wavelength λ_0 at the entrance slit width of 10 μm is equal to $\lambda/\delta\lambda$=250-1000 depending on the grating and the wavelength λ_0. The spectrographs have a removable cassette for a photographic film allowing one to record several spectra without breaking vacuum.

Depending on the vacuum chamber installation and the heights of spectra, three variations are possible:

GIS-1: The height of a spectrum at a focal plane is 20 mm. The spectrograph is installed inside the vacuum chamber in a standard 2 inches or 60 mm optical mount. This variation has very small sizes—a diameter of 45 mm and a length of 180 mm (minimal λ_0) - 300 mm (maximal λ_0). The device is installed close to the radiation source (up to ~10 mm).

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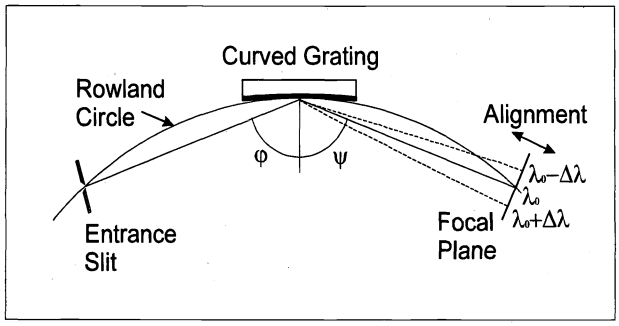


Fig. 1. The scheme of the grazing incidence spectrograph (GIS).

GIS-2: The height of a spectrum at a focal plane is 30 mm. The spectrometer is connected to the vacuum chamber by an adapter flange and has a port for a differential pump. This device has a diameter of 90 mm, a length of 180 mm (minimal λ_0) - 300 mm (maximal λ_0).
GIS-3: The height of a spectrum at a focal plane is 40 mm. The spectrometer is connected to the vacuum chamber by an adapter flange and has a port for a differential pump. The device has a diameter of 130 mm and a length of 180 mm (minimal λ_0) - 300 mm (maximal λ_0).

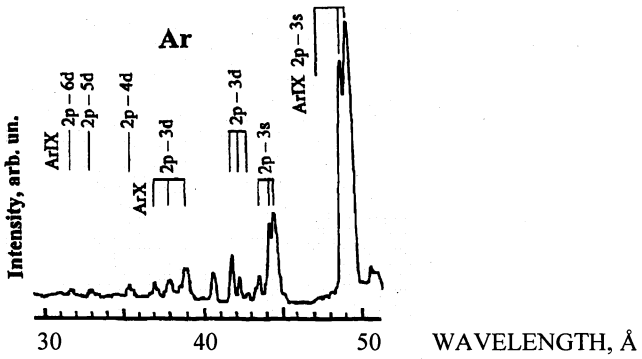


Fig. 2. Spectrum of Ar IX – Ar X ions in Ar filled capillary. [3]

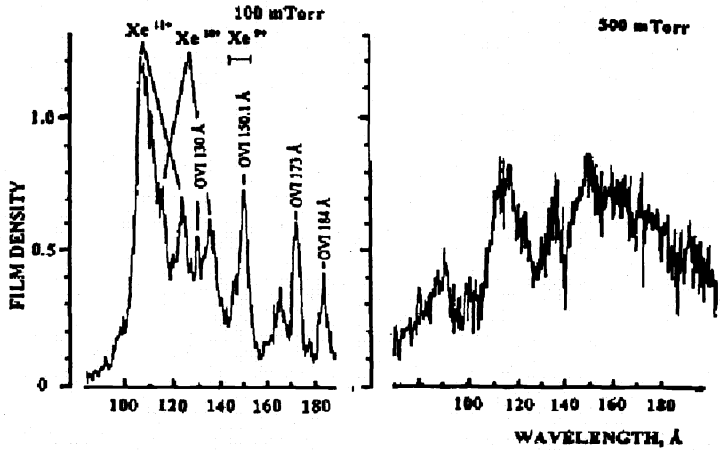


Fig. 3. Time- integrated axial emission spectra in Xe gas-filled capillary discharges at different gas pressures (100-500 mTorr). [3] Xenon-ion stages contributing emission are indicated. Oxygen – contamination lines are also noted.

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The spectrographs were used for EUV spectroscopy of a KrF laser plasma source, intended for EUV lithography, [1] and for EUV spectroscopy of capillary discharge plasmas. [2, 3] Spectra from various target materials produced by a KrF-laser were investigated in EUV spectral range between 12 and 17 nm. The electron temperature, ionization stages and conversion efficiency at 13.5 nm were measured from the spectra.[1] The spectrograph was used for investigating EUV spectra (spectral range of 30-200 Å) of fast capillary discharge plasmas. [2, 3] Strong linear emission of highly charged ions from a polyacetal capillary and from the gas-filled capillary was observed. The spectra were investigated for different capillary geometries and various gas pressures. Examples of time-integrated axial emission spectra in Ar and Xe gas-filled capillary discharges[3] are shown in Figs. 2 and 3.

3. GRAZING INCIDENCE MONOCHROMATOR – SPECTROMETER (GIMS)

For some practical applications (for example, in reflectometry) it is necessary to form a monochromatic EUV beam in a constant direction. For this purpose a versatile grazing incidence spectrometer with constant angle of deviation, α, was developed (see Fig. 4). The wavelength scan is carried out by a precise rotation of the diffraction grating. The parameters of the spectrometer are chosen in such a way that the defocusing due to radiation falling outside the Rowland circle was small and did not worsen the spectral resolution. The angle of deviation is equal to $\alpha=\varphi+\psi=166°$. The angle of incidence, φ, and diffraction angle, ψ, can be changed as $\varphi = 85° \pm 1°$ and $\psi = 81° \pm 1°$. The angles are changed, and alignment on different central wavelengths λ_0 is achieved by using a precise grating rotation. In the spectrometer three diffraction gratings are used (see Table 1). Each grating is mounted in its individual pre-aligned holder. The spectral range, covered by the three gratings, is 44.3 – 425.2 Å. The spectral range can be extended in both sides with some losses in spectral resolution or realignment of the exit slit position. The spectrometer has micrometer driven entrance and exit slit assemblies. The slit width can be varied from 0.5 μm to 0.2 mm with an accuracy of ~ 0.5

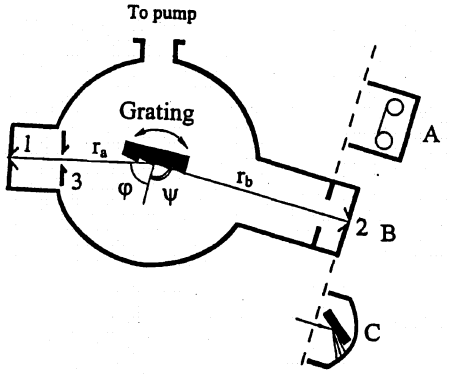


Fig. 4. The scheme of the grazing incidence spectrometer-monochromator (GIMS): 1 – entrance slit; 2 – exit slit; 3 – diaphragm; φ - incidence angle; ψ - diffraction angle; $\varphi+\psi=166°$; r_a – distance between entrance slit and grating; r_b - distance between the grating and exit slit; A – spectrograph mode; B – monochromator mode; C – reflectometer mode.

μm. The slit height is 20 mm. Some parameters of the spectrometer are listed in Table 1: recommended spectral range, dispersion D (Å/mm) in the exit slit plane, spectral resolution $\delta\lambda$ at the entrance slit width of 15 μm. The device has a port for differential pumping. The spectrometer is compact—spectrometer length ~ 250 mm.

The spectrometer can be used in three modes: spectrograph, monochromator and reflectometer. In the spectrograph mode a removable cassette for photographic film is installed instead the exit slit (monochromator mode). In this mode the exact focusing takes place at only one central wavelength λ_0 near the exit slit position. However due to the small numerical aperture of the spectrometer, it is possible to record the spectrum in a wide spectral range $\lambda_0 \pm \Delta\lambda$. The value $\Delta\lambda$ is related to the observed spectral resolution $\lambda/\delta\lambda$ - the wider $\Delta\lambda$, the lower $\lambda/\delta\lambda$ due to the defocusing. The alignment for different wavelengths, λ_0, is produced by a grating rotation.

In the reflectometer mode a special chamber is installed behind the exit slit. Using this chamber, the reflection coefficient of grazing incidence gratings and mirrors can be measured.[4]

This spectrometer was used for EUV spectroscopy of gas-filled capillary discharge plasmas and reflectometry purposes.[4,5] The use of various gases (CO_2, Ne, Ar, Kr, Xe) allows the observation of EUV spectra in a wide spectral range – 4 – 45 nm. As an example spectra in Ne and Kr gas-filled capillary discharges are shown in Figs. 5 and 6.

The spectrometer with a known grating reflectivity and absolutely calibrated detectors [6] becomes an absolutely calibrated device. It has been used for the measurement of absolute emission yield from Xe gas filled capillary near the wavelength of 13.5 nm – the region of interest for EUV lithography applications. [4,5]

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Table 1. Parameters of the grazing incidence spectrometer: recommended wavelength range, dispersion D (Å/mm) in the exit slit plane, spectral resolution $\delta\lambda$ at the entrance slit width of 15 μm.

Grating, G/mm	Wavelength range, Å	D (Å/mm)	$\delta\lambda$, Å
1200	44.3 – 106.3	7.3-9.1	0.5
600	88.6 – 212.6	14.6-18.2	1.0
300	177 – 425.2	29.2-36.4	2.0

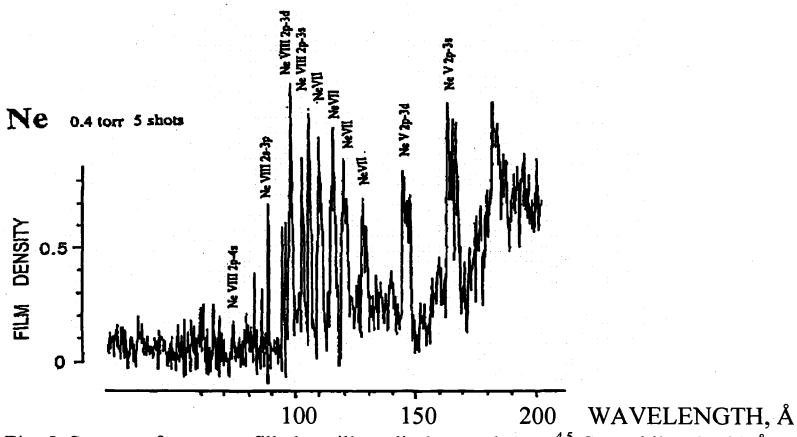


Fig. 5. Spectra of neon gas filled capillary discharge plasma.[4,5] Central line λ_0=90 Å.

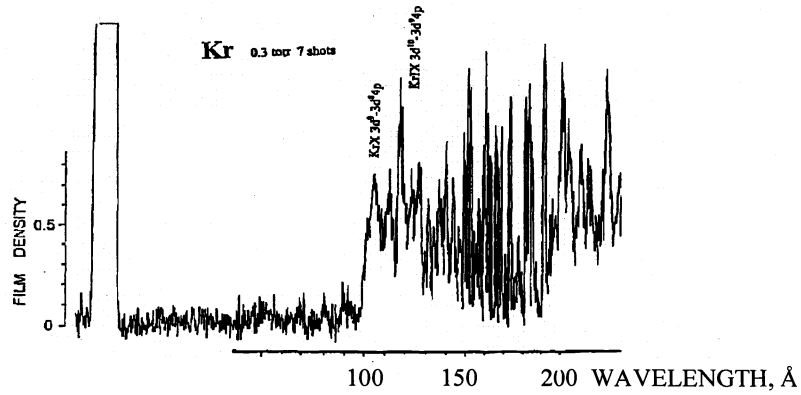


Fig. 6. Spectra of krypton gas filled capillary discharge plasma.[4,5] Central line λ_0=165 Å.

4. FOCUSING CRYSTAL VON HAMOS SPECTROMETER

The maximum efficiency of x-ray spectroscopic devices can be achieved when focusing crystal x-ray spectrometers and solid state detectors are used. Currently, charge coupled devices (CCD) are often used as active recording detectors in soft x-ray imaging systems. CCD x-ray detectors have the advantages of high sensitivity, good signal-to-noise ratio, wide dynamic range, and good spatial resolution.

The use of a CCD as a detector in the focusing scheme of crystal x-ray spectrometers can increase the sensitivity of the device. One of the focusing schemes of crystal x-ray spectrometers is a von Hamos scheme.[7, 8] In the von Hamos scheme, a crystal is bent into a cylindrical surface. The x-ray source and the detector plane lie on the cylinder axis (see Fig. 7). Radiation of each wavelength, after diffraction from the crystal arc, is focused to a point lying on the spectrometer axis. This focusing geometry gives high spectrometer efficiency—focusing intensities could be 10^2-10^3 times higher than those of flat crystal spectrometers.[8] Another distinctive feature of the von Hamos geometry is that it allows the use of mosaic crystals with highly integrated reflectivity. This increases spectrometer efficiency further. In this case, mosaic focusing in the direction of dispersion takes place. This results in high sensitivity without sacrificing spectral resolution.[9]

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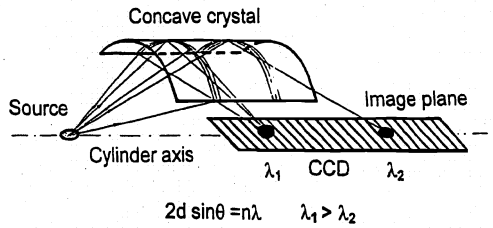


Fig. 7. The scheme of the von Hamos spectrometer.

In the von Hamos geometry x-ray spectra are formed on the spectrometer axis. This makes it possible to use a CCD linear array as an x-ray detector. The large length of the detector (in our case 30 mm) makes it possible to record x-ray spectra in a wide spectral range.

In this work a commercial CCD linear array was used as absolutely calibrated detector in the focusing crystal von Hamos spectrometer. The spectrometer was used for absolute spectral measurements of x-ray yield and for determination of parameters of laser-produced plasmas.[10, 11] This compact device has a very high detection efficiency in a wide spectral range and can be used for absolute spectral measurement of x-ray radiation from low intensity sources and in numerous practical applications (XRF analysis, EXAFS, etc.).

The von Hamos spectrometer was used for absolute x-ray spectral measurements of laser-produced plasmas. [11] Laser plasmas were created by focusing of the "Phoenix" Nd glass laser radiation (0.53 μm/10 J/2 ns) onto massive solid targets (Mg, Al, Ti, or Fe).

The commercial CCD used for x-ray detection is the Toshiba model TCD 1304AP. It is a linear array with 3724 pixels. Each pixel is 8 μm wide (6 μm active) and 200 μm high. The total effective detecting area is 200 μm by 29.8 mm (8 μm x 3724 elements). The CCD works at room temperature.

The sensitivity of the CCD linear array was absolutely calibrated in the spectral range of 2-10 Å using laser-produced plasmas and Fe^{55} isotope source. [10, 11] The CCD is 140-840 times more sensitive than the photographic film Kodak RAR 2492.

A mica crystal ($2d$=19.84 Å) and highly oriented pyrolytic graphite (HOPG, $2d$=6.708 Å)[12] with a radius of curvature R=20 mm were used in the von Hamos spectrometer. The crystal integrated reflectivity was measured earlier using laser-produced plasmas,[8, 9] an Fe^{55} isotope source and a MOXTEK silicon PIN diode detector. [9] The CCD linear array was positioned in the image plane (the spectrometer axis) with a tolerance of (±20 μm). To prevent visible light illumination, a double Al-coated mylar filter (4.4 μm of mylar, 0.2 μm of Al) was used. Spectra were recorded in a singe laser shot.

The x-ray spectra of highly charged ions excited in laser-produced plasmas were recorded for the first, third and fifth orders of mica reflection and for the first order of graphite reflection. The measured crystal integrated reflectivity [8, 9] together with the CCD absolute calibration data, allowed us to record the spectra on an absolute intensity scale. As an example an x-ray spectrum of a Fe laser-produced plasma in the vicinity of a resonance line of the helium-like ion is shown in Fig. 8.

X-ray spectra obtained with the mica crystal show high spectral resolution—the spectral resolving power $\lambda/\delta\lambda$ was equal to 800, 2000 and 2000 for Mg (first reflection order), Ti (third reflection order) and Fe (fifth reflection order) plasmas, correspondingly. In the spectrum of Ti plasma recorded by the graphite crystal, the spectral resolving power is lower—$\lambda/\delta\lambda$=200-300. Nevertheless, due to mosaic focusing this value is higher than $\lambda/\delta\lambda$=50 defined by the mosaic spread of the graphite crystal.[9]

The high spectral resolution of the mica crystal von Hamos spectrometer allowed us to observe the complete structure of the x-ray spectra—resonance lines of the hydrogen- and helium-like ions and their satellite lines. These spectra were used for determination of plasma parameters—electron temperature T_e and density N_e—using x-ray spectroscopy methods.[13]

Due to the focusing geometry of the von Hamos scheme and high efficiency of the CCD detector the spectra were observed at very low laser pulse energies (as low as 14 mJ for Mg plasmas, see Fig. 9). In most cases of spectra recording it was necessary to apply severe attenuation to the incidence intensity (up to 20000 times) to avoid CCD saturation. For this reason this spectrometer is very good for studying the x-ray spectra of low intensity sources—femtosecond laser-produced plasmas, micropinches, EBIT sources, etc.

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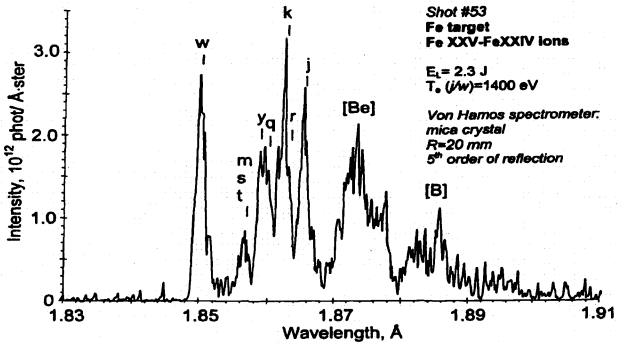


Fig. 8. Spectrum of a Fe laser-produced plasma in the vicinity of resonance line *w* of [He]-like ion. [13]

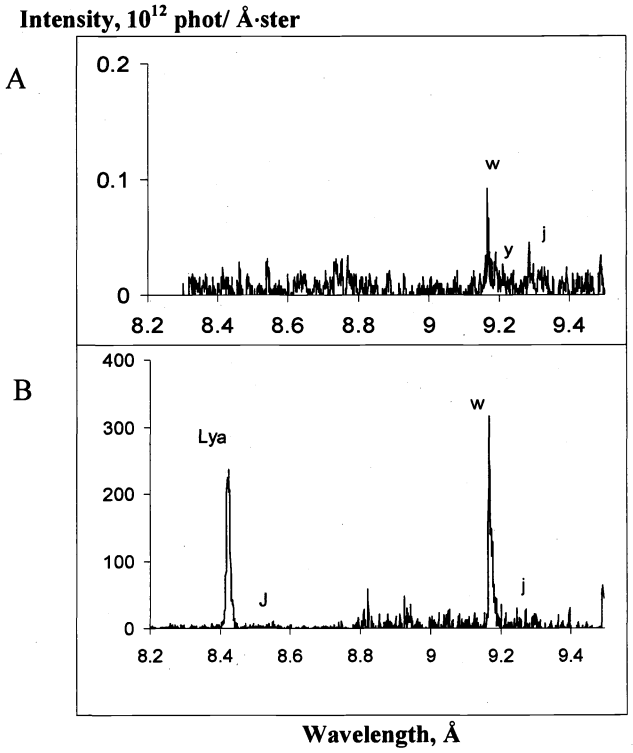


Fig. 9. Spectra of Mg laser-produced plasmas in the vicinity of resonance lines Ly_α and *w* of hydrogen-like and helium-like ions, respectively. A – the laser pulse energy E_L=14 mJ, electron temperature T_e=220 eV, B – the laser pulse energy E_L=0.5 J, electron temperature T_e=550 eV.

5. FOCUSING CRYSTAL VON HAMOS SPECTROMETERS FOR XRF APPLICATIONS

In order to increase the energy resolution in X-Ray Fluorescence (XRF) devices, it is necessary to use crystal-based spectrometers. The von Hamos spectrometer is very suitable for this purpose. This type of spectrometer has the following definite advantages over other focusing schemes made it useful in XRF—high efficiency in a wide spectral range (or energy range); high spectral resolution (in our case $E/\Delta E$=50-100); simplicity; wavelength (or energy) scans are accomplished by a simple linear translation of a detector; the possibility to use mosaic crystals to increase efficiency without sacrificing spectral resolution; and compact size. These features make it possible to use the von Hamos spectrometer device in XRF applications.[14]

In Ref. [14] an excellent use of the spectrometer to determine the levels of sulphur in oil and other petroleum product is described. Sufficient energy resolution is required to separate lines such as the S K_α from the Pb *M* lines (ΔE approximately 30 eV), yet there is a limited set of elements that is of analytical interest.

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Two von Hamos spectrometers with mica and graphite crystals (20 mm radius of curvature) were design and built. Absolute efficiency of the spectrometers was measured at wavelength of about 2 Å using Fe^{55} isotope source. An x-ray tube with Ag anode in a transmission mode was used as an excitation source (Model BS-1, Svetlana-Rontgen, St. Petersburg). Operating conditions were 25 kV and 0.3 mA emission current, 7.5 watt power. A MOXTEK energy-dispersive PIN diode detector (MOXTEK PIN diode with 8 μm DuraBerllium™ window) was used to reduce the contribution of scattered light from sample. The detector was movable within the spectrometer in mounting sleeve.
Minimum detection limits for 10 minute collection times were determined. The spectrometer with mica crystal has demonstrated 14 ppm sensitivity for sulphur in oil (at standard concentration of 1000 ppm). The Hamos multidispersive spectrometer with graphite crystal has demonstrated 2 ppm sensitivity for sulphur in oil (at standard concentration of 100 ppm). In the last case with reduced background and scatter the spectrometer should be capable of better than 400 ppb detection limit for sulphur. In addition to sulphur, a moveable detector will allow analysis for lead, vanadium, iron and other elements.
Future enhancements will include the use of miniature x-ray tubes[15] to reduce the size of the spectrometer and the use of CCD detectors for simultaneous collection of energy-dispersive spectra and spatial resolution information.

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