High resolution short focal distance Bent Crystal Laue Analyzer for copper K edge x-ray absorption spectroscopy

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We have developed a compact short focal distance Bent Crystal Laue Analyzer (BCLA) for Cu speciation studies of biological systems with specific applications to cancer biology. The system provides high energy resolution and high background rejection. The system is composed of an aluminum block serving as a log spiral bender for a 15 micron thick Silicon 111 crystal and a set of soller slits. The energy resolution of the BCLA—about 14 eV at the Cu Kα line— allows resolution of the Cu Kα_1 and CuKα_2 lines. The system is easily aligned by using a set of motorized XYZ linear stages. Two operation modes are available: incident energy scans (IES) and emission energy scans (EES). IES allows scanning of the incident energy while the BCLA system is maintained at a preselected fixed position—typically CuKα_1 line. EES is used when the incident energy is fixed and the analyzer is scanned to provide the peak profile of the emission lines of Cu. © *2011 American Institute of Physics*. [doi:10.1063/1.3595675]

I. INTRODUCTION

X-ray absorption spectroscopy (XAS) has become an essential tool for coordination chemistry.[1] This technique allows evaluation of an element's oxidation state and local coordination environment by determining its absorption spectrum. Many research fields have incorporated this powerful technique including material science,[2] geological and environmental sciences,[3] chemistry,[4] and biology.[5]

While transmission measurements are the standard mode of operation for concentrated samples, fluorescence emission measurements are the preferred mode when dealing with dilute samples. In the latter case, the fluorescence x-rays emitted by the sample are detected by energy dispersive or wavelength dispersive detectors. Solid state detectors are very popular as energy dispersive systems (EDS) because of their small size, easy operation, and very good efficiency. Their energy resolution, typically around 160 eV at Mn Kα line, allows resolution of the fluorescence lines of the element of interest from other fluorescence lines and background signal. Wavelength dispersive systems (WDS), on the other hand, offer much higher energy resolution in the order of a few eVs, but they are in general less practical because they require to be attached to a goniometer for proper positioning and their efficiency is very low compared to solid state detectors (in a WDS system about 30% of the x-rays entering the detector are actually counted; whereas, in an EDS system about 100% of the incident x-rays are counted depending on the energy range). In recent years an alternative approach for wavelength dispersive detectors has been proposed by applying thin crystal analyzers bent in a log spiral configuration.[6–8] The so called Bent Crystal Laue analyzers (BCLA) are commonly used in synchrotron facilities showing important advantages over conventional detection systems when applied to the study of diluted systems, although conventional BCLA devices are not able to resolve Kα_1 and Kα_2 spectral lines.

There are many biological systems, including single cells,[9] proteins,[10] and tissues sections,[11] that are amenable to investigation using XAS techniques. A variety of elements including Fe, Mn, Cu, and Zn play important roles in the biological functionality of the systems under study. Because metal concentrations in biological samples are, in general, in the few parts per million range, fluorescence emission measurement is the preferred mode of operation. Due to the low concentration of the elements of interest in biological samples they usually require a high flux incident x-ray beam to produce sufficient fluorescence signals. A significant amount of scattering signal is also generated because of the light element matrix constitution of the samples. Furthermore, it can be very difficult to study a system that contains more than one elemental species that have very similar spectral features.[12]

The high background contribution from the light matrix combined with the low fluorescence signal may result in the signal from the low concentration metal elemental species being masked by the background, making their identification very difficult. This problem becomes more acute with energy dispersive detection systems because they collect all the incident photons that reach the detection area increasing the dead time of the system significantly with the risk of saturating the detector. The advantage of BCLA based detection systems for these cases is that they can reject with high efficiency the unwanted photons from scattering signal and other fluorescence lines and offer high energy resolution.

Copper (Cu) is an essential trace metal for all mammals, including humans.[13] Cu is known to be a cofactor of angiogenesis, i.e., the growth of a tumor blood supply, an essential prerequisite for tumor growth, invasion, and metastasis.[13] Cu

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levels and chemical status might be implicated as a characteristic of the tumor growth process. The study of Cu level and oxidation state in human prostate and breast tissue by x-ray absorption spectroscopy could provide important information relating Cu accumulation, its chemical status, and the tumor progression stage.

In this paper, we describe a recently designed compact Bent Crystal Laue Analyzer with short focal distance from sample that provides high energy resolution and detection efficiency for copper speciation studies of biological systems with specific applications to cancer biology. The short focal distance allows the collection of sufficient fluorescence signal from dilute samples due to its large solid angle acceptance. The energy range of the analyzer is centered at the Cu Kα line for Cu speciation studies. Since the energy resolution of the BCLA is a function of the incident beam size, we used an x-ray microprobe as the excitation source to obtain near theoretical resolution allowing the Cu Kα_1 and Cu Kα_2 lines to be distinguished. The detection system is completed by set of soller slits and coupled silicon drift detector (SDD) that works as a photon counter. This configuration allows collection of only the Cu Kα photons with very low background contributions because the analyzer offers a very high background rejection ratio.

II. ANALYZER DESIGN

The conceptual design of the BCLA system relies on the logarithmic spiral bending of a thin crystal to resolve the mismatch between the narrow acceptance angle of a perfect crystal and the large divergence of fluorescence photons emitted from the sample. Previously, a BCLA device reported by Zhong *et al.*[6] showed that this type of bending could be used for x-ray spectroscopy determinations for energies above 13 keV although their device lack soller slits that could reject the unwanted transmitted beam. Later, Kropf *et al.*[8] developed a BCLA that incorporated soller slits, improving energy resolution, but not sufficient to resolve Kα_1 and Kα_2 lines in that energy range. Zhong's and Kropf's devices were limited to higher energies because the absorption of the fluorescence photons by the ~140–200 microns thick crystal used. The need of thinner crystals for low energies down to 5 KeV was proposed by Kropf *et al.*[8] but not reported before. Our device was designed for thin crystals to cover a lower energy range near Cu K lines.

In general, the main advantage of the BCLA geometry is the enhanced reflectivity width due to the bending of the lattice planes in the transmission geometry. The most important characteristic of BCLA is that the crystal is bent in a logarithmic spiral shape which solves the mismatch between the narrow acceptance angle of a perfect crystal and the large divergence of fluorescence photons from the sample. We have used the REFLECT program[14] developed to predict crystal reflectivity. In polar coordinates, a logarithmic spiral is represented by the following equations:[6–8]

$$r(\theta) = \rho_0 \cos(\chi - \theta_B) \exp[\tan(\chi - \theta_B)\theta], \quad (1)$$

$$\frac{\Delta E}{E} = \Delta\theta(T)\cot(\theta_B), \quad (2)$$

where ρ_0 is the bending radius of the crystal wafer at the center $\theta = 0$, θ_B is the Bragg angle of the wavelength of interest, and χ is the crystal asymmetry angle between the reflecting planes and the surface normal. The diffracted beam is deflected by angle $2\theta_B$. For matching the Bragg condition over a bending angle, the energy resolution is given by Eq. (2), $\Delta\theta(T)$ is the bandwidth and T is the crystal thickness. We used computer programs written in Interactive Data Language (IDL, Research Systems, Inc. Boulder, CO) and Mathematica (Wolfram Research, Inc. Champaign, IL) to solve Eqs. (1) and (2) to determine the proper crystal shape for the energy range of interest.[15] Our calculations allowed us to design a compact BCLA with 5 cm focal distance and energy range centered at the Cu Kα line using a 15 micron thick silicon crystal (diffraction plane: [100] surface oriented Si wafer and ⟨111⟩ reflection planes of Silicon crystals were used). Although bending such a thin crystal might be expected to distort the crystal planes, this effect was not large enough to prevent our device from providing high energy resolution at the Cu K emission lines. Our results, given below, are showing that the use of thin crystal is practical for obtaining high energy resolution at relatively low x-ray energies. This is the principal finding of our study and has not been reported before.

The calculated reflectivity of current BCLA is 35% and the asymmetry angle is 35.26°. Figure 1 shows the front and side views of the analyzer. Because accurate bending is difficult to achieve dynamically, we used an aluminum block bender that provides the correct log spiral shape. Even though the analyzer can be mounted in any orientation relative to the beam direction, it is typically mounted in a standard 45 degree angle configuration. The block has a 45 degree angle cut on the left side down (see Fig. 1) that allows placement of the analyzer close to the sample.

Because the 15 micron thick crystal transmits a large fraction of the photons reaching the crystal surface (one absorption length for Si is 66 microns at Cu Kα line and 93 microns at 9 keV incident energy), a key component of the Laue analyzer is the set of soller slits used to eliminate the unwanted contribution of the transmitted signal (see Fig. 2(a)). The soller slits slots, spaced at 3 mm each, were machined on
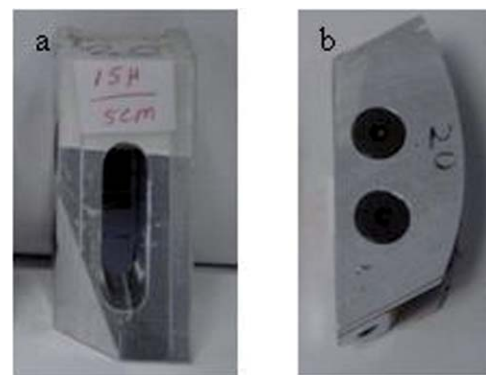

FIG. 1. (Color online) (a) Front view of the BCL analyzer. The 45 degree angle cut, at left-down on the picture, allows moving the BCLA near the sample that is located at 45 degree angle relative to the incident beam. (b) Side view of the aluminum block where the hole for a 1/4-20 screw used for mounting purposes can be seen on the bottom.
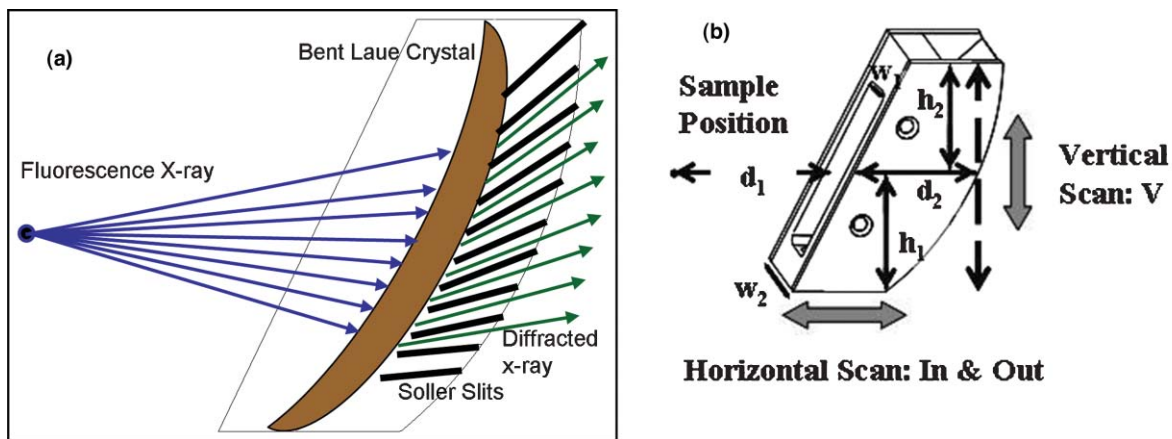
FIG. 2. (Color online) (a) Sketch of the logarithmic bent Laue crystal analyzer. The x-ray fluorescence source is located about 5 cm from the surface of the crystal at the BCLA. The blue color lines represent the fan-like distribution of the fluorescence x-rays, brown color represents the bent crystal (not in scale), thick black lines are the soller slits and green arrows represent the direction of the diffracted fluorescence lines; and (b) sketch of the two motions required for the alignment of the BCLA: V for energy alignment and In&out for focal distance alignment. Dimensions of the analyzer are shown in the drawing: $d_1 = 38$ mm, $d_2 = 24$ mm, $w_1 = 8$ mm, $w_2 = 20$ mm, $h_1 = 22$ mm, $h_2 = 18$ mm. Focal distance is determined between source position and crystal surface, a_1 distance is shorter than focal distance.

the aluminum block such that they have the same angular divergence of the diffracted beam—thus absorbing the transmitted beam and any incident photons diffracted at other angles. The chosen slits are 10 mm long and made of molybdenum (Mo). Molybdenum was chosen because of its high absorption coefficient at energies near the Cu Kα line and because its K absorption edge (@ 20 KeV) cannot be excited by the scattered x-rays. This greatly improves background rejection and enormously simplifies construction of the devices and experimental alignment. The combination of a thinner crystal, a short bending radius, and a smaller micron size beam spot provided the required resolution to discriminate Cu Kα_1 and Cu Kα_2 lines.

The efficiency of this BCLA detector system at higher energy decreases because the shallow incidence angle needed to preserve the Bragg condition reducing the angular acceptance. As a consequence, this detector system is best used at low energies (between 7.5 and 9.5 KeV). A large active area silicon drift detector (Ketek V450R100-ZR5BE 160), used as photon counter, completes the setup. The fluorescence signal of the Cu Kα x-rays diffracted by the BCLA is collected by the SDD located behind the soller slits. A region of interest around the Cu Kα line is selected at the XIA multi-channel spectrometer to further improve the signal to noise ratio. This system proved to have very high background rejection with high energy resolution and sufficient efficiency for Cu speciation studies.

Another important feature of our device is that we use a set of linear stages for alignment and scanning purposes. Previously reported devices[6–8] required a goniometer to align the analyzer making the alignment process much more complicated. Additionally, because of the limited energy resolution of these devices, none of the previous studies reported on the emission peak profile. Additionally, we used a SDD as counting device to improve our signal/noise ratio. Any unwanted photons that could reach the detecting area are later rejected by the very narrow region of interest defined on the MCA. Even though this could result in a reduction of the total

active area of the analyzer, this did not detract from our goal of obtaining sufficiently high energy resolution with very good signal to noise ratio for the detection of very low Cu concentrations in tissue samples.

III. EXPERIMENTAL

A. Microprobe beamline components

As mentioned previously, the best performance of the BCLA system is obtained when using a micro beam as the source of x-rays because the energy resolution of the BCLA is a function of the incident beam size, especially in the vertical direction. Undulator-based x-ray microprobes are clearly the best choice for incident x-rays sources because they can provide high flux and small size x-ray beams. For this purpose we take advantage of the existing microprobe instrument at the undulator-based BioCAT beamline 18ID at the Advanced Photon Source (APS), Argonne National Laboratory (ANL). The BioCAT microprobe optics consist of a cryogenically cooled Si (111) double-crystal monochromator and a pair of 200 mm long Kirkpatrick-Baez (KB) Rh-coated mirrors, located 70 m downstream of the undulator source. The beam is micro-focused using the KB mirrors[16] which provide a focal spot of ~5 micron FWHM and flux of 1.3×10^{12} photons/s at 10 KeV. The intensity of the microprobe x-ray beam is monitored using an ionization chamber located immediately downstream of the KB mirrors. A detailed description of the beamline and its microprobe-associated instrumentation has been presented elsewhere.[17,18]

B. BCLA alignment

The complete system composed of the crystal bender, soller slits, and the SDD is mounted on a fixed support which is attached to a kinematic mount to allow reproducible mounting of the device on an XYZ motorized stage. One of the advantages of the current design is that it does not require a goniometer to rotate the crystal for energy selection. Instead, a

set of three high-precision linear stages are used to provide the horizontal, vertical, and focal positioning of the BCLA.

The horizontal stage allows one to center the analyzer's aperture with the beam at a 90 degree angle, which is maintained fixed during the experiments. The focal positioning stage allows motion of the BCLA toward the sample position so the beam-analyzer distance corresponds to the focal distance. The vertical stage ("energy stage") allows selection of the energy to be detected by the SDD. The x-ray microprobe was set initially at incident energy of 9.2 KeV for BCLA alignment purposes. The alignment process involves a two-dimensional scanning of the focal and vertical stages while measuring the Kα line emitted by a Cu sample as shown in the Fig. 2(b). The final focal position is determined based on the energy resolution observed during the scanning of the vertical stage. Once the optimum focal distance is found, it remains constant for the remainder of the experiment.

For calibration and alignment purposes we used the following samples: Cu foil, CuO, Cu_2O, CuS, and $CuCl_2$. The samples had a thickness about 50–100 microns to minimize self-absorption effects. Samples are placed at 45° relative to the incident x-ray beam in a standard fluorescence configuration. The sample positioning is performed by a second set of high precision (0.1 μm resolution) XYZ motorized stages.

C. BCLA scans

The BCLA system provides two modes of operations: incident energy scans (IES) and emission energy scans (EES). By scanning the incident energy provided by the monochromator while maintaining the BCLA "energy stage" at a fixed position, we can obtain the standard x-ray absorption spectrum in fluorescence mode. We call this mode of operation IES. In this mode of operation, the BCLA "energy stage" is at a fixed position to allow detection of the intensity of the selected fluorescence line, typically Cu $K\alpha_1$. EES are performed by maintaining the incident energy (provided by the monochromator) constant while the BCLA "energy stage" is scanned to detect the profile of the emission lines. The main advantage of these EES scans is that the intensity from the fluorescence peak is determined with minimal background contribution, even when the incident energy is close to the absorption edge.

IV. RESULTS

A. Energy resolution

The BCLA energy resolution is determined by performing EES scans as described in Sec. III C. Figure 3 shows the $K\alpha_{1,2}$ fluorescence spectra of a thin Cu foil sample measured with the BCLA in EES mode. The analyzer yields an energy resolution of 14 eV at the Cu Kα line. As demonstrated in the figure, this resolution is sufficient to resolve the Cu $K\alpha_1$ (8.046 keV) and $K\alpha_2$ (8.026 KeV) lines which are separated by $\Delta E = 20$ eV. The shoulder on the high energy side of the fluorescence peaks is due to imperfections on the bender block that cause a deviation from the ideal bending shape of the Si(111) crystal. For comparison, the fluorescence
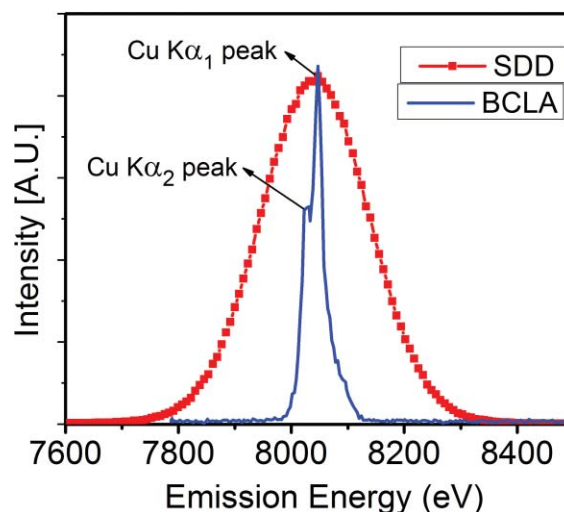

FIG. 3. (Color online) Cu K Fluorescence lines determined using the BCLA with 9.2 keV incident energy. CuKα_1 and CuKα_2 are clearly resolved. Same spectral line determined using a silicon drift detector is shown for comparison purposes. Notice the high energy resolution of the BCLA compared to the SDD.

spectrum of the same sample using a Si drift solid state detector is also shown.

B. Detection limit

One of the advantages of the low-background contributions to the signals collected by the BCLA is the improved low detection limit. The detection limit of a system is typically defined as the minimum detectable amount of a given element present in a sample and it is directly related to the background level under the peak of interest. In the case of the BCLA, the background level is significantly reduced by the high rejection ratio provided by the combination of the soller slits and the region of interest windows defined on the spectrometer. We have determined a calibration curve using Cu diluted samples prepared following standard laboratory procedures. We determined that the minimum detection limit for this system is 5 ppm of Cu (see Fig. 4). The acquisition time per sample was 30 s.
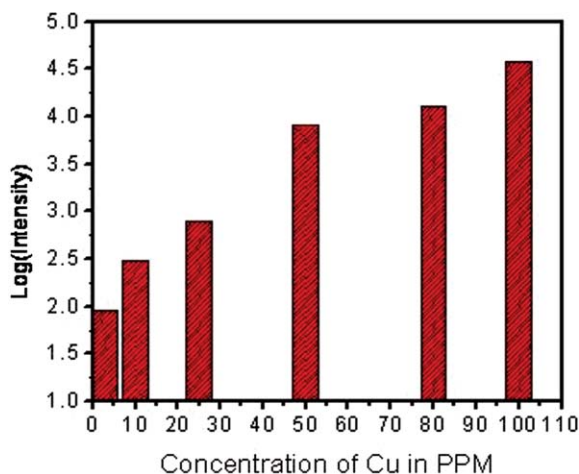

FIG. 4. (Color online) Sensitivity curve determined using NIST Cu standards. Minimum detectable Cu level observed is 5 ppm of Cu.
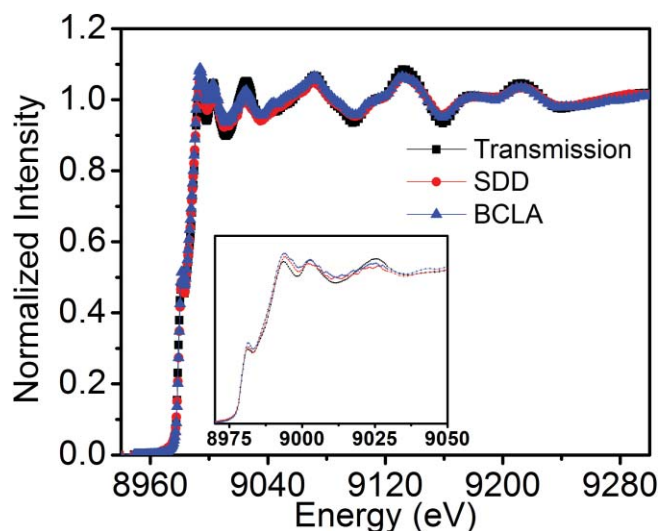
FIG. 5. (Color online) X-ray absorption spectrum measured at Cu K edge of a Cu foil sample determined using the BCLA. Same spectrum measured in transmission mode and in fluorescence mode using a SDD are shown for comparison purposes. Some self-absorption effects are observed in the fluorescence spectra due to the thickness of the foil sample. These effects are usually negligible when measuring biological samples because the sample thickness is typically 20–30 microns thick and the Cu concentration is in the order of a few ppm.

C. Cu K edge XANES determinations

X-ray absorption near edge structure (XANES) measurements at the Cu K edge were performed with the BCLA system set at the Cu $K\alpha_1$ peak. For comparison purposes, we also performed XANES measurements in transmission mode using two ion chambers, and in standard fluorescence mode using a solid state SDD. Figure 5 shows the measured data obtained with each measurement mode: transmission, fluorescence with SDD, and fluorescence with BCLA. It can be seen that the fluorescence spectra obtained with either the SDD or the BCLA fall directly under the transmission data although some reduction of the oscillation signal is observed due to the self-absorption effects affecting the Cu foil sample. It is expected that this effect will be greatly reduced when working with tissue sections or very thin samples (dilute samples). The XANES scans in fluorescence mode were taken in continuous scan mode covering a 300 eV range with a 0.5 eV step size and a scan time of 30 s. For the SDD system alone, 30 scans are typically required to achieve an acceptable counting statistics yielding a total of 900 s of scan time. This acquisition time increases proportionally with the level of dilution of the sample. The main disadvantage observed is that the scattering contribution from the matrix could be considerably large, increasing the dead time of the detection system. In this case, it is recommended to reduce the flux delivered or increase the sample-detector distance to reduce the dead time to acceptable values. For the BCLA, the number of scans increases to
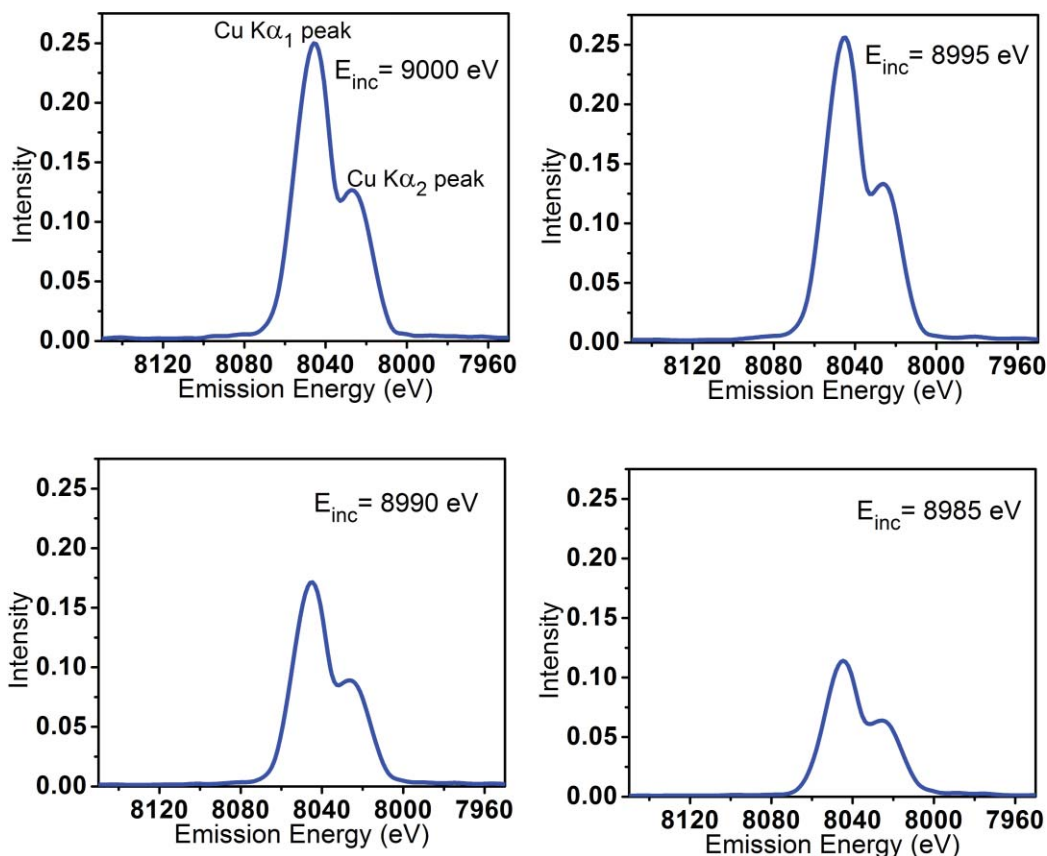

FIG. 6. (Color online) EES BCLA scans of Cu K lines at four different incident energies. Notice the energy resolution allows resolving clearly the $CuK\alpha_1$ and $CuK\alpha_2$ lines. Also notice the low background level. Each scan requires only 30 s.
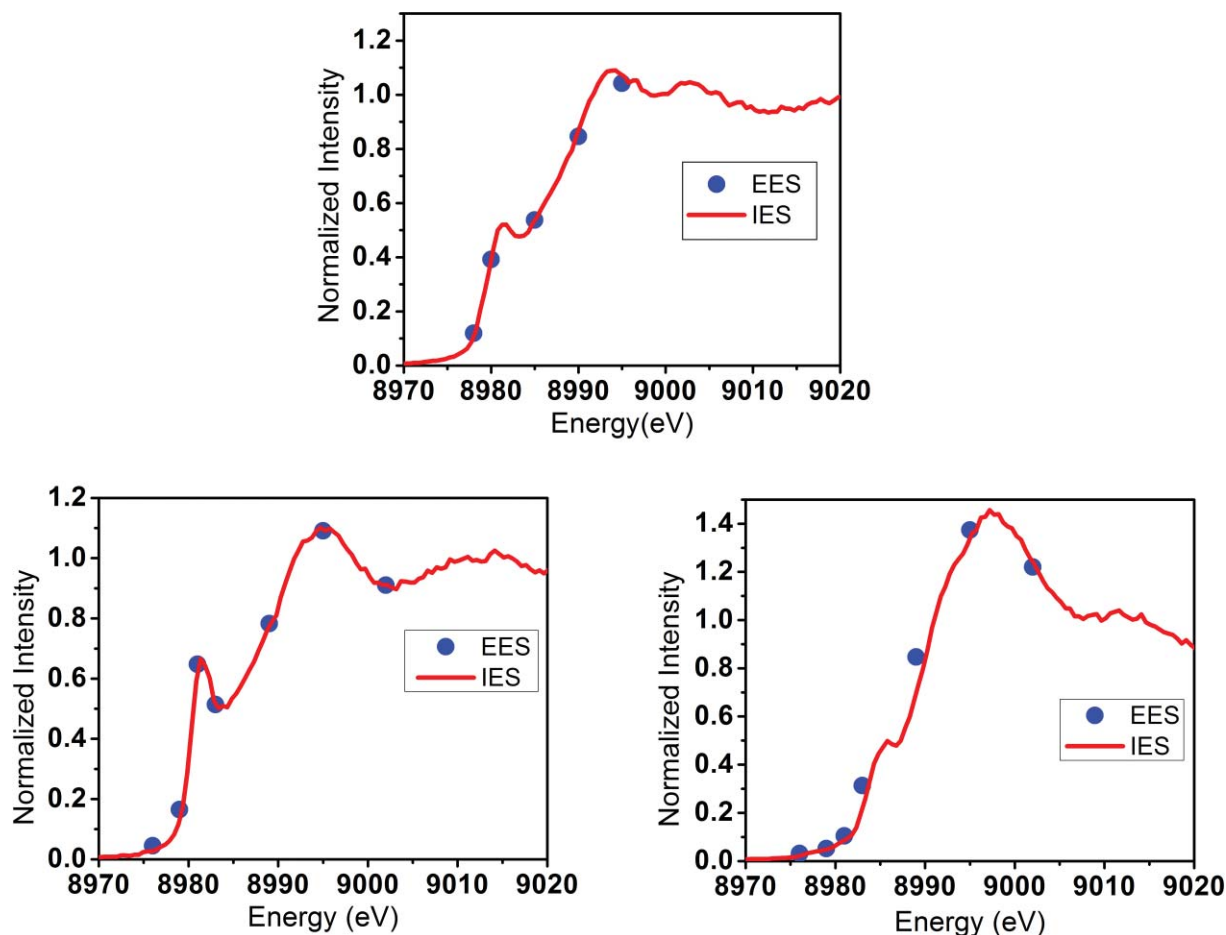
FIG. 7. (Color online) (Top) Cu foil incident energy scans (IES, solid line) and EES scans (EES, dots) obtained with the BCLA analyzer; (bottom left) Cu(I) standard compounds same experimental conditions; and (bottom right) Cu(II) standard compound spectra.

100 or more because of the low counting rate observed. In this case, the total scan time is about 3000 s. The reduction of the background contributions outweighs the reduction in count rate for the fluorescence signal resulting in a better signal to noise ratio.

D. Peak profile determinations

As described in Sec. III C, the BCLA system can be scanned in emission energy scan mode, EES, to determine the peak profile of Cu Kα line. We performed EES scans at four pre-selected incident energies: 8985, 8990, 8995, and 9000 eV to observe the profiles of the Cu Kα_1 and Cu Kα_2 lines. Figure 6 shows the EES spectra obtained. The measured Cu Kα_1 and Kα_2 lines were fitted using Gaussian shape functions. The integral value of the area under these peaks can be used to reconstruct the intensities of the Cu line under the IES scan by integrating the peak profile and normalizing the intensity to a standard value. The intensity is then assigned to the corresponding energy pixel in the IES scan spectrum. Figure 7 shows the resultant integrated values for a set of pre-selected incident energies represented as dots while the IES scan data are represented by a continuous line. As observed in the graph, the EES values follow the same pattern as the IES scans. This comparison proves that XAS spectra can be obtained using the BCLA in EES scan mode.

V. CONCLUSIONS

We have developed a compact short focal distance Bent Crystal Laue Analyzer for Cu speciation that provides high energy resolution and high background rejection. The system is composed of an aluminum block serving as a log spiral bender for a 15 micron thick Silicon 111 crystal and a set of soller slits. A silicon drift detector used as a counter completes the arrangement. The energy resolution of the BCLA— about 14 eV at the Cu Kα line—is dependent on the incident beam size, therefore a microprobe is needed to achieve best performance. The detection system is easily aligned using a set of motorized XYZ linear stages. Two operation modes are available: IES and EES. IES allows scanning of the incident energy while the BCLA system is maintained at a preselected fixed position—typically the CuKα_1 line. EES is used when the incident energy is fixed and the analyzer is scanned to provide the peak profile of the emission lines of Cu. The energy resolution of the system allows resolution of the Cu Kα_1 and CuKα_2 lines.

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