CONF-8808141--4 BNL--41811

Beam Line X-11A at the NSLS: A Unique Facility for X-ray Absorption Spectroscopy

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BNL--41811

DE89 002177

ABSTRACT

The design and operation of beam line X-11A at the NSLS is described. It employs a unique optical design using a two/four crystal monochromator and a SiC collimating mirror. In either two or four crystal mode a sagittal focusing crystal allows collection of up to 5 mrad of horizontal divergence. Two techniques for rapid scanning of a sagitally focusing crystal will be described and compared. These are dynamically bending the crystal during the scan or translating a fixed radius bent crystal in a manner to maintain a fixed focus on the sample. The flexibility of this two/four crystal design allows the intensity and/or resolution of the beam line to be optimized for particular experiments.

1. Introduction

X-ray beam lines for use with EXAFS experiments must satisfy a number of conflicting requirements. The beam lines must provide good energy resolution and reproducibility, high intensity, good beam stability, and be rapidly tunable. In addition, it is often desirable to provide a focused beam for small samples. To satisfy these requirements a unique facility has been constructed on beam line X-11A at the National Synchrotron Light Source (NSLS).[1] It combines a two or four crystal monochromator with sagittal focusing and a collimating mirror. The set up is very flexible allowing the beam line configuration to be optimized to the experiment.

2. Beam Line Design

Figure 1 shows the basic layout of the beam line. Since this line is designed for operation at high energies (4-30 keV), the use of focusing mirrors would place a severe restriction on the amount of radiation which can be collected. For practically realizable mirror lengths an ellipsoidal or toroidal mirror can collect only 2 mrad at 30 keV. However, sagitally focusing crystals have been demonstrated to be capable of collecting up to 10 mrad in this energy range.[2]

For a small source such as the NSLS, significant intensity gains can be achieved with a collimating mirror. In this case only the vertical divergence need be collimated and practical mirrors can easily collect 10 mrad or more of the horizontal divergence. We have chosen to use a spherical approximation to be a parabolic surface for such a collimating mirror preceding the monochromator. Unfortunately, fabrication problems and shipment damage have precluded testing of the mirror to date.

2.1 Monochromator

Figure 2 shows the basic components of the monochromator. It is based on two Huber 421 goniometers. Each goniometer has a pair of crystals and piezoelectric adjusters for fine tuning the parallelism of the crystals. The second goniometer is mounted on a one meter translation stage for use with sagittal focusing. This will be discussed in the next section. The entire instruement is housed in a vacuum tank and operates at $\simeq 10^{-5}$ torr. Not shown in the drawing are Pb collimators for blocking line of sight radiation.

Since this drawing was made, the second and third crystals have been replaced with large fixed crystals which increase the energy range and allows the third crystal to be water cooled. This was necessary because when operating in two crystal mode the beam is allowed to strike the third crystal directly by raising the entire monochromator. This can be done without breaking vacuum and the change over including alignment can be accomplished in about 1/2 h.

Currently the angle range of the monochromator is 5-35° which gives an energy range of 3.5-22 keV with Si(111) crystals. The addition of larger second and third crystals could decrease the lower limit to 3.5° allowing operation to $\simeq 30$ keV. The energy resolution of the Si(111) crystals is well matched to the intrinsic resolution of absorption edges in this energy range and they are the preferred crystals when operating in four crystal mode. For two crystal operations a set of Si(311) are being prepared for the high energy range.

2.2 Optical Principles

Sagittal focusing using perfect crystals has recently been developed by Sparks and co-workers.[2] Some of the basic principles are given here.

Consider first two flat crystals in a nondispersive (+,-) configuration. Each ray striking the first crystal makes the same angle on the second crystal giving $\Delta\theta=\theta_2-\theta_1=0$. If the second crystal is bent to a radius R in the sagittal direction then to lowest order

$$\Delta\theta = \frac{\omega_h^2 F_1}{R} - \frac{\omega_h^2 F_1^2}{2R^2} \sin\theta_o$$

where ω_h is the horizontal divergence of the ray from the central axis, F_1 is the distance to the source, and θ_o is the incidence angle of the central ray. From the above it is seen that $\Delta\theta=0$ when

$$R = \frac{F_1 \sin\theta_o}{2} \quad .$$

The lens equation for sagittal focusing gives

$$R = \frac{2F_1 F_2}{F_1+F_2} \sin\theta_o$$

where F_2 is the crystal to focus distance. Thus, for $F_2=F_1/3$, $\Delta\theta=0$ and the throughput of the focusing monochromator is equal to the non-focusing flat crystal arrangement.

To maintain the focusing condition as the energy is scanned it is necessary to vary the crystal radius. This is an option with our monochromator, using a simple crystal bender, but may be difficult for more complicated benders capable of collecting large horizontal angles. Therefore, an additional translation capability was designed into the monochromator. Keeping R fixed, F_1 and F_2 can be varied such that the focal distance $L=F_1+F_2$ is fixed as θ is changed. If the translation is small, $\Delta\theta$ remains small (less than the crystal rocking curve) and there is little loss in intensity. To lowest order in the angle change δ

$$\Delta\theta = \frac{2\omega_h^2 \delta}{3 \sin^2 \theta_o} \quad .$$

Thus, the outer most rays are lost first as δ is increased. Figure 3 shows the intensity variation with energy scanning for various energies and crystals for our beam line design when 10 mrad of radiation is collected. This result was obtained by ray tracing and includes source size effects along with calculated diffraction profiles from perfect crystal theory. It is seen that for typical EXAFS scans of ~1000 eV the intensity variations are <20% in most cases. The intensity variations can be easily reduced if necessary by reducing ω_h.

The energy scan range available for fixed R depends on the translation travel. For a source to focus distance of L the variation in F_1 is

$$F_1 = \frac{L}{2} + \frac{L}{2} \left(1 - \frac{3}{4} \frac{E}{E_o}\right)^{1/2} \quad .$$

For our monochromator design the translation stage of 1 m allows energy scans of 1 keV for $E_o = 6$ keV increasing to 3.3 keV scans at $E_o = 20$ keV.

3. Operation

The monochromator has been operating extensively in both two and four crystal modes without sagittal focusing. Since the collimating mirror for the beam line has not been installed, much of the operation has been in two crystal mode. This offers higher intensity and adequate resolution for most applications when a collimating mirror is not used. This mode is common to many monochromators and will not be discussed further here. Since four crystal operation and sagittal focusing are more unusual, more details are given.

3.1 Four Crystal Operation

One problem of running with four crystals is the initial alignment. Three elements must be aligned before any x-rays are observed: the two crystal pairs must each be aligned and the two goniometers must be set to equal angles. Therefore, it is essential that provisions be made for making these three adjustments independently. The second crystal pair can be aligned by operating in two crystal mode. The first pair is aligned using an internal ion chamber or by observing a fluorescent screen. Once the individual pairs are aligned it is then a simple matter to scan one goniometer until an output beam is observed in four crystal mode. In our case the full alignment procedure is not always necessary. The crystals are pre-aligned on gimbal mounts to ~50 arcsec accuracy. This is sufficient to produce a very small but measurable signal, and alignment can then proceed by maximizing the intensity.

The second problem is maintaining alignment during scanning. The Huber goniometers have periodic errors of ≈30 arcsec with a one degree period. To correct for these an analog feedback circuit is used. When operating without a collimating mirror this feedback is based on beam position rather than beam intensity. The beam striking the first pair of crystals is ≈20-30 arcsec wide while the monochromator only passes a few arcsec of beam. For maximum intensity this should be the central portion of the incoming beam. However, since the beam shape is approximately Gaussian, deviations of ≈1-2 arcsec about the central maximum have little affect on intensity, but 1 arcsec corresponds to 80 μm of motion at the sample. When attempting to measure an absorption signal to 10^{-4} accuracy it is often difficult to prepare a sample uniform enough to tolerate such motion. Therefore, the best solution was found to be a system which used a split ion chamber beam position monitor, and attempts to keep the beam position constant. The accuracy of the current system is about ±20 mm. This is adequate for most experiments, except those requiring the utmost beam stability such as glancing angle EXAFS studies. For these two crystal operation is preferred. It is planned to replace the current split ion chamber with one of greater sensitivity. This may allow 4 crystal operation for essentially all samples.

Figure 4 shows some examples of the energy resolution obtainable with four crystal operation using Si(111) crystals. No entrance slits were used. These scans also demonstrate the success of the feedback system in that the monochromator did not need to be re-aligned in moving between these edges except for a simple detuning of the second crystal pair to remove harmonics. As can be seen the energy resolution is quite good approaching the intrinsic resolution of the edges involved. In moving over these large angular ranges it was found helpful to synchronize the two monochromator motors. This minimizes the errors with the feedback system has to correct, and allows it to always remain locked onto the beam during movement.

One final characteristic of the position based feedback system is worth mentioning. It also acts to remove beam position fluctuations due to source movement. The current system can remove fluctuations up to about 5 Hz although a faster HV amplifier could push this limit to much higher frequencies. The position accuracy is only limited by the accuracy of the position monitor.

3.2 Focusing

Initial testing has been carried out using a focusing crystal capable of collecting about 5 mrad of radiation. Figure 5 shows an example of the focused beam. The unfocused beam is seen to have stripes. These are due to the grooves cut into the face of the crystal to avoid antielastic bendings.[2] In our case, we chose to work with thin (0.75 mm) Si wafers. These were grooved with a 0.05 mm diamond saw using standard Si processing equipment.[3] The crystal has a triangular shape and is bent using an Inchworm[4] translator. The main part of the focused beam is ≈2x2 mm and the intensity is $\approx 2.5\times10^{10}$ photons/sec (normalized to 100 ma ring current). This is about a factor of two less than the expected intensity. The reduction is most likely due to imperfections in cutting the grooves, leading to nonuniform bending of the crystal.

Tests of energy scans with the focused beam have recently began. Figure 6 shows a scan of the Fe edge using the bent crystal. In particular, Fig. 6(b) shows the variation of I_o during the scan. I_o is seen to vary smoothly. Most of the fall off at higher energy is due to the decay of the ring current, since this scan was made during the recommissioning of the NSLS ring and the beam lifetime was short.

Currently, the beam line software is being modified to support dynamic crystal bending. Initial tests indicate that this is also a feasible mode of operation. It has the advantage that once the bender is calibrated, changing energy does not require user intervention.

4. Conclusions

The beam line presented has a number of unique characteristics which allow it to be optimized for particular EXAFS experiments. It can be run in two crystal or four crystal mode depending on whether intensity or energy resolution is the important factor, and the addition of a sagittally bent crystal allows focusing to high energies. The design allows scanning with focusing to be carried using either a fixed radius crystal or by dynamic bending. The utility of the beam line has resulted in more than 100 publications during the first two years of operation using flat crystals, and with the addition of focusing the beam line should become even more useful.

Acknowledgements

This work is supported by the auspices of the U.S. Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences under Contract Nos. DE-AS05-80-ER10742 and DE-AC02-76CH00016.

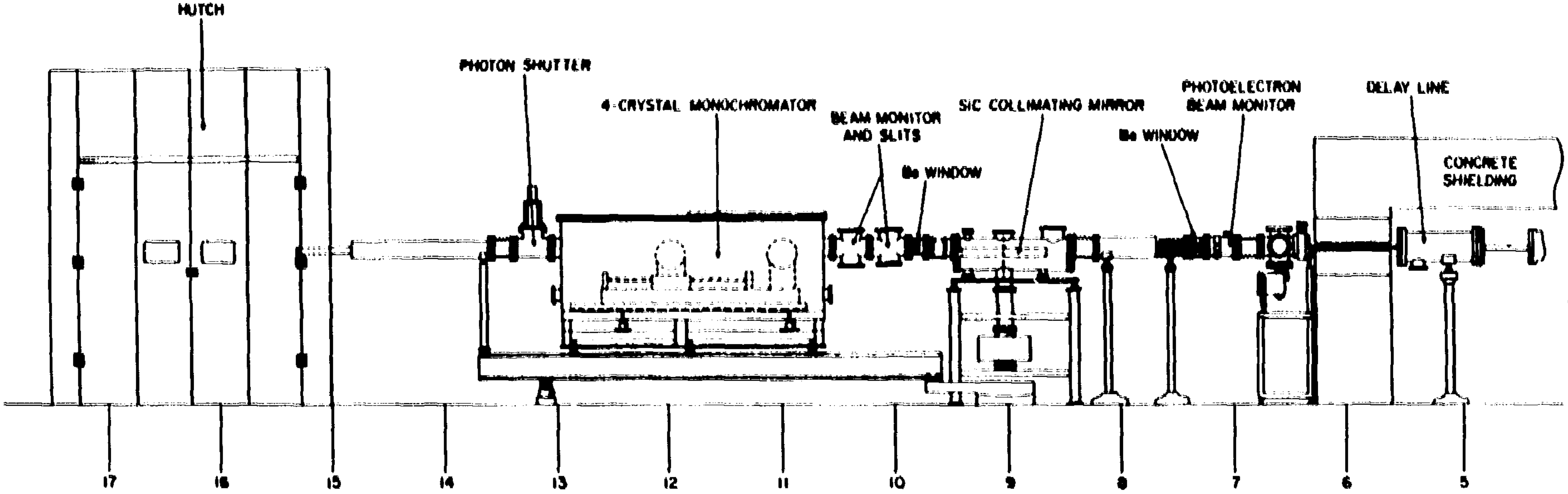
References

1. See also D. E. Sayers, S. M. Heald, M. A. Pick, J. I. Budnick, E. A. Stern, and J. Wong, Nucl. Instrum. Methods <u>208</u>, 631 (1983); S. M. Heald, Nucl. Instrum. Methods <u>A266</u>, 457 (1988).
2. C. J. Sparks, G. E. Ice, J. Wong, and B. W. Batterman, Nucl. Instrum. Methods <u>194</u>, 73 (1983).
3. The crystals were fabricated by Semiconductor Processing Company, Boston, MA.
4. Burleigh Instruments, Fishers, NY.

Figure Captions

1. Beam line schematic.
2. Schematic of the monochromator: CB, crystal bender; CT, crystal translation stage; EN, encoder; GC, goniometer carriage for 1 m translation; GM, gimbal mount for pre-aligning crystal; H, Huber model 421 goniometer; MM, motorized micrometer for coarse alignment; PZ, piezoelectric translator for fine crystal alignment; SM, stepping motor; WC, water cooled crystal. Not shown is piezoelectric adjuster for adjusting the angle of the first goniometer.
3. Intensity variations with scanning for a fixed radius bent crystal collecting 10 mrad. The curves are for 5, 10, 15, and 20 keV. The solid lines are for Si(111) and the dashed line for Si(220) crystals.
4. Edge scans for Cu (edge energy = 8980 eV), $KMnO_4$ (6555 eV) and anatase TiO_2 (4980 eV). The monochromator was operated in four crystal mode with the entrance slit wide open.
5. Photograph of the unfocused (top) and focused (bottom) beams. The actual width of the unfocused beam is ≈8 cm, with the photograph being limited by the exit window of the beam line.
6. (a) EXAFS scan of a 6 μm Fe foil using a focused beam. The crystal radius is fixed and the monochromator is translated to maintain a fixed focus.

   (b) I_o intensity for the scan in (a).


HUTCH
PHOTON SHUTTER
4-CRYSTAL MONOCHROMATOR
BEAM MONITOR AND SLITS
Be WINDOW
SiC COLLIMATING MIRROR
Be WINDOW
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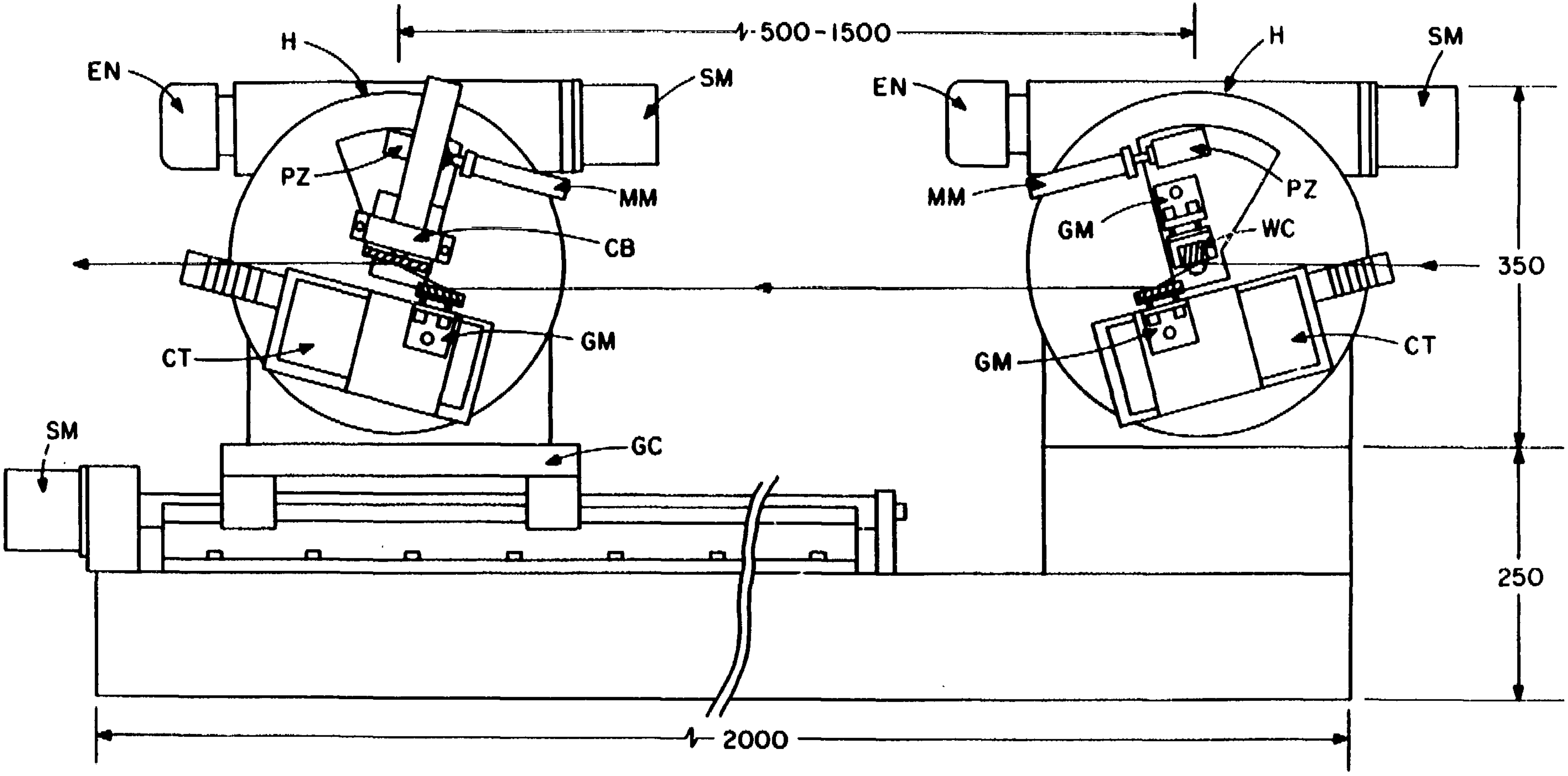

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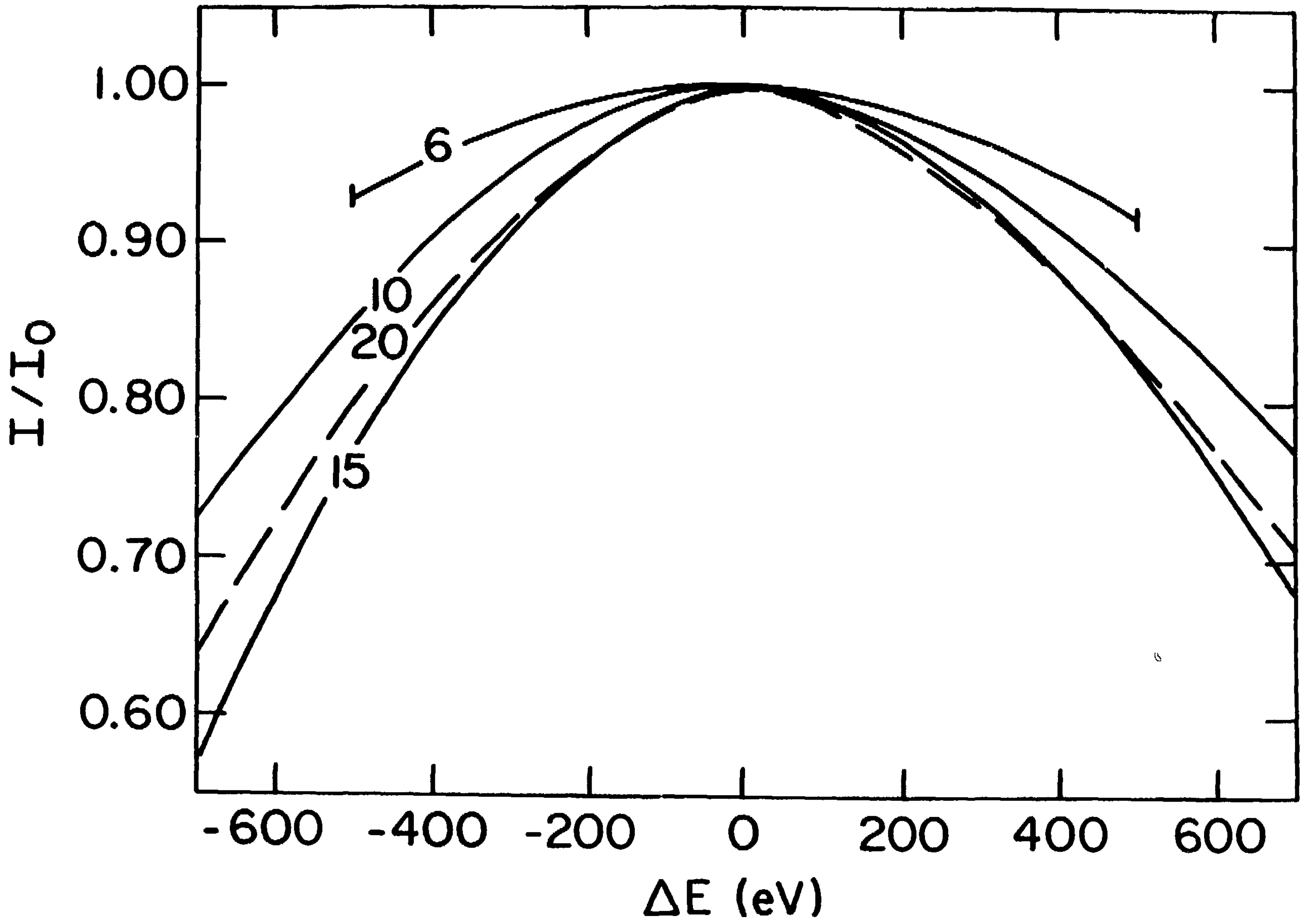
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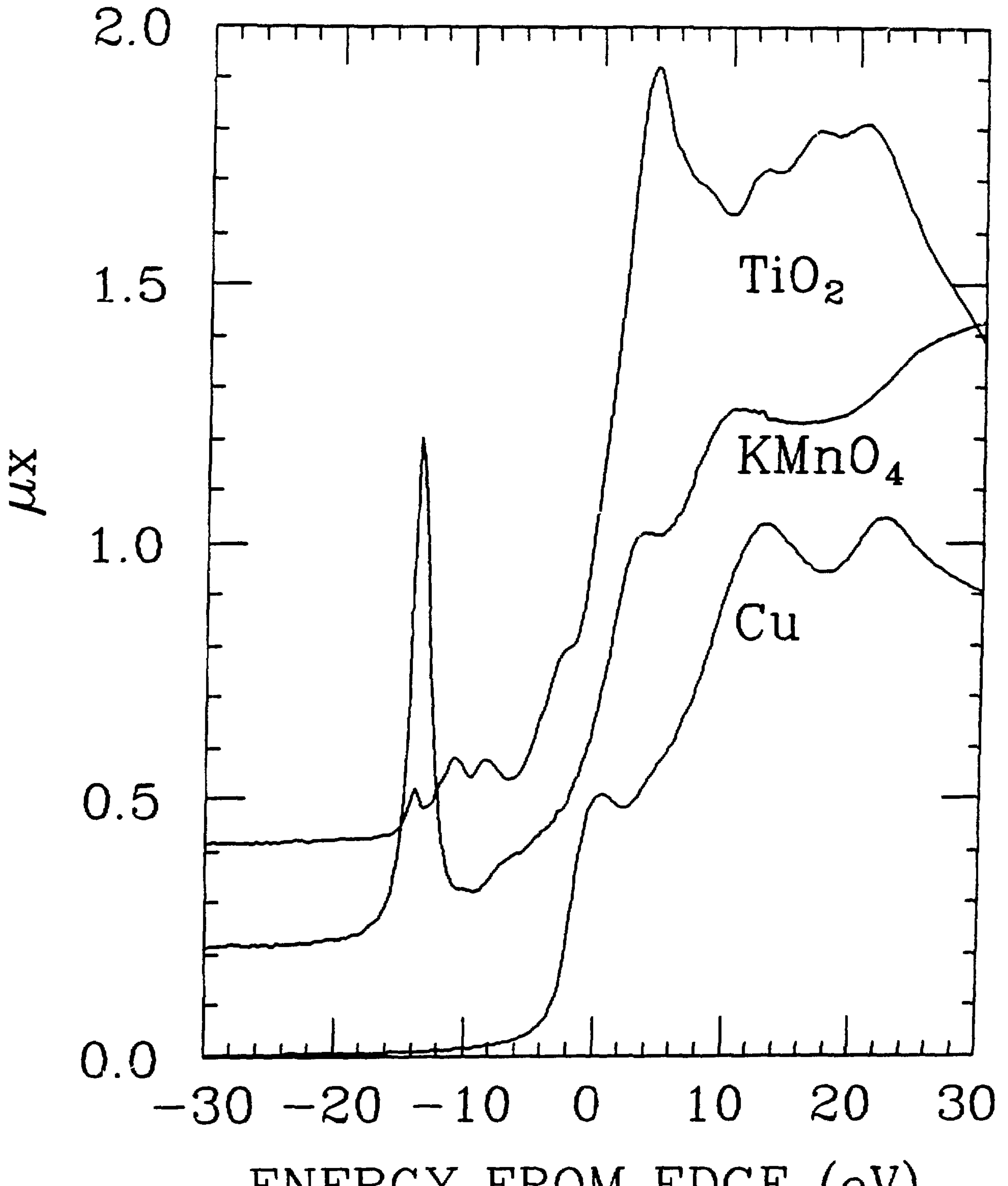
ΔE (eV)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μx
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ENERGY FROM EDGE (eV)

Fig 2

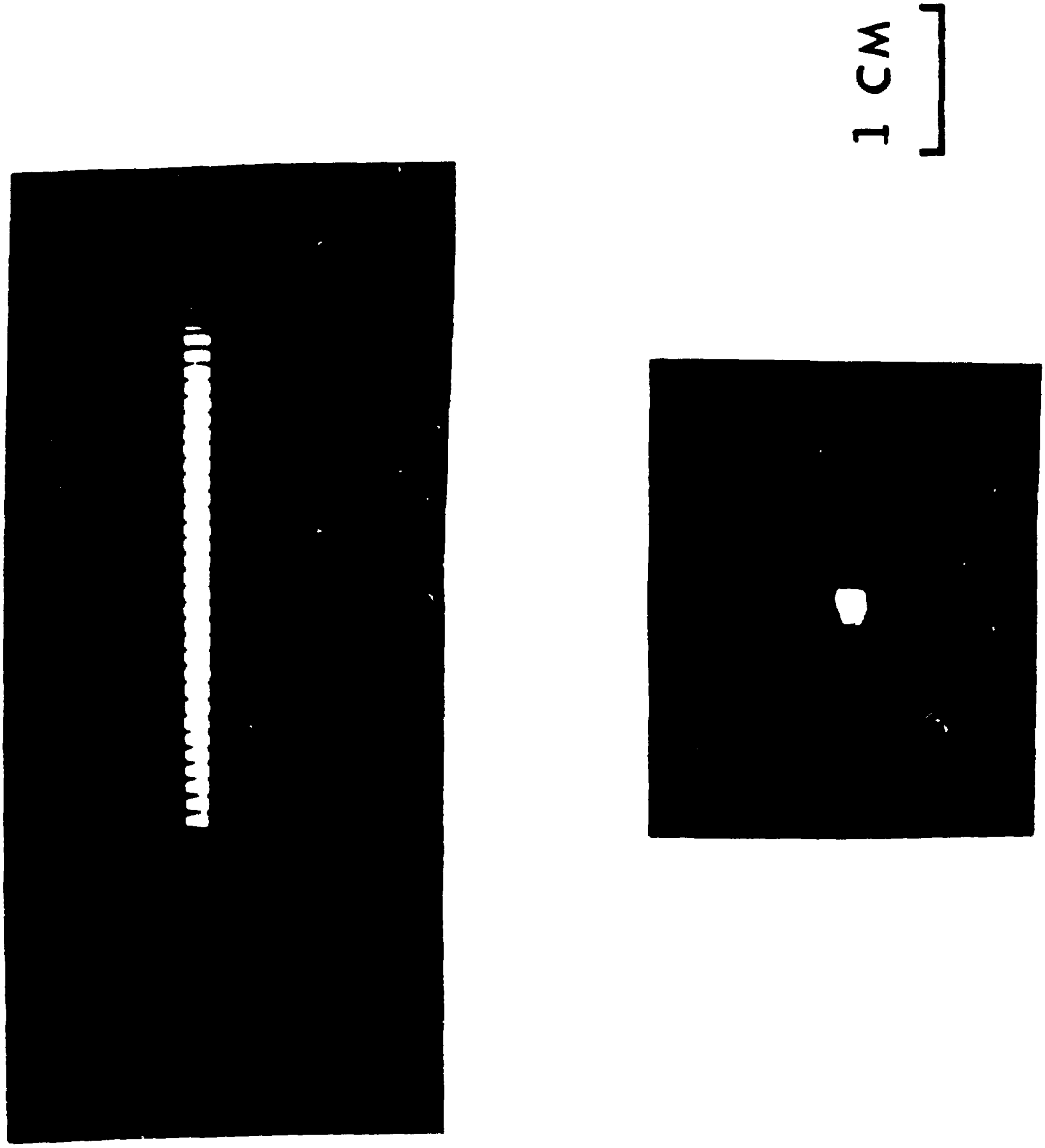
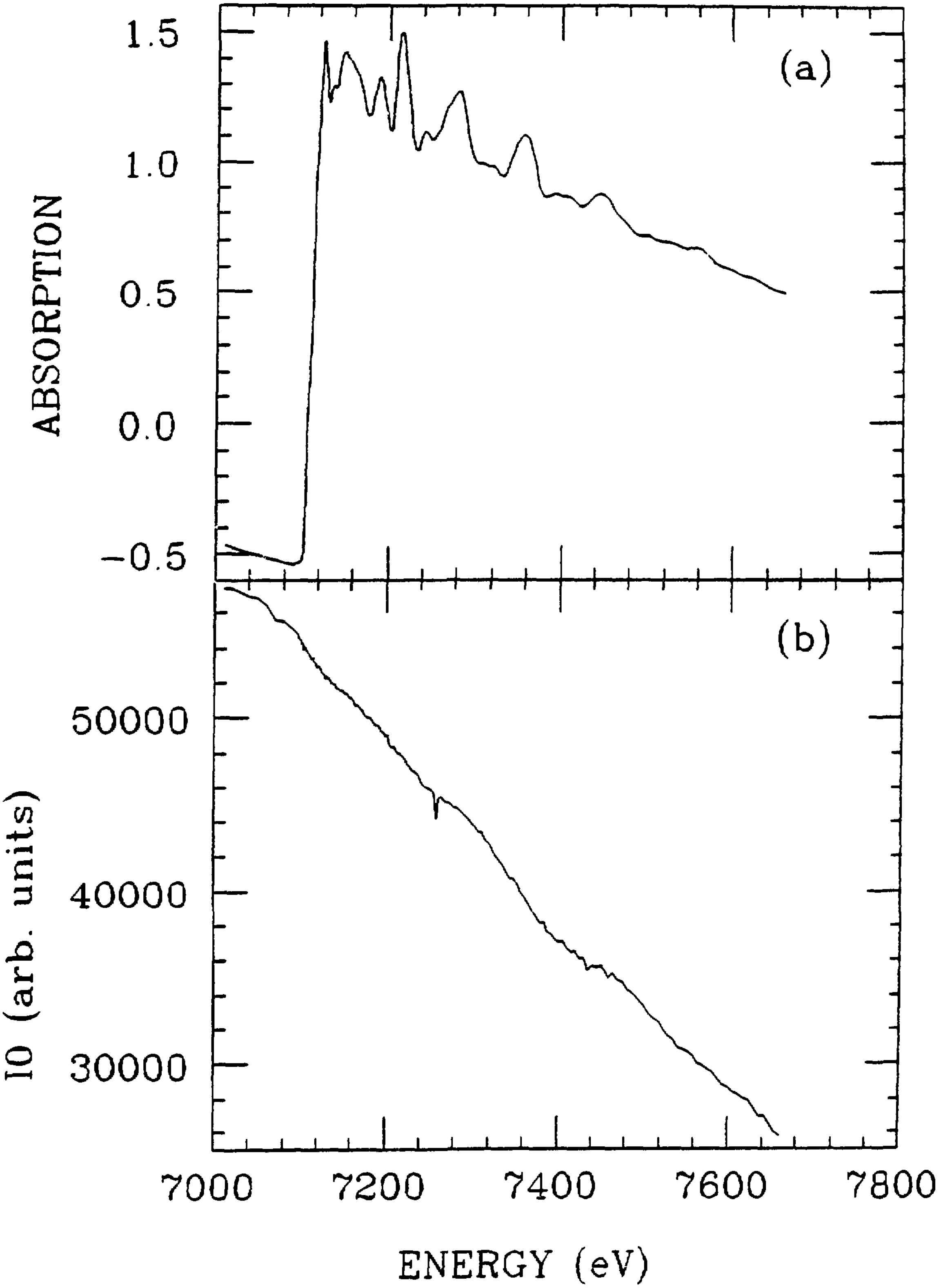


Fig 5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ENERGY (eV)

Fig 6