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MEMORANDUM

To: Distribution
From: M. E. Glinsky MEG
Subject: Measurement of up to 0.2 eV Crystal Resolution at 870 eV

Attached is a paper describing the work done to date on the measurement of sub-volt crystal resolution at 870 eV using a neon gas absorption cell illuminated by the nickel L_β line.

MG:lr

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Lawrence Livermore
National Laboratory

Neon K-edge as a Tool to Measure Crystal Resolving Power at 870 eV

by
M. E. Glinsky

ABSTRACT

The neon 1s-3p absorption line at 866.9 eV is illuminated by the Ni L_β line at 868.8 eV. This is made possible by the relatively broad width of the L_β line (~7 eV) compared to the narrow neon 1s-3p absorption line (~0.2 eV). The absorption line is measured by both a curved and flat mica crystal (plane 002). The width of the measured absorption line indicates that the resolving power of the flat mica crystal is 0.72 ± 0.14 eV at 870 eV. The calculated resolving power using the Darwin-Prins dynamical theory of X-ray diffraction is 0.52 eV. The results for the curved crystal resolution are inconclusive because of lack of source intensity.

INTRODUCTION

The measurement of the resolving power of crystals requires that the beam of X-rays be made monochromatic at a certain angle to an amount greater than the resolving power of the crystal. This is commonly accomplished by use of an analyzing crystal. When the resolution of the crystal which needs to be measured is better than or equal to any other crystal at the wavelength of interest this becomes impossible. The best which can be done is to use two crystals of the same type. This provides a rough estimate of the width of the crystal rocking curve, but all information on the shape of the crystal curve is lost. A further complication arises at sub-kilovolt energies because the absorption of the crystals becomes larger than the extinction due to diffraction: the peak reflectivity decreases from 100% to less than 2% for mica at 870 eV. When two crystals are used the intensity is decreased a factor of 50 or greater over what it would be for a single crystal.

The solution to this problem is to use a source with an energy feature narrower than the resolving power of the crystal. If the collimation is narrower than the width of the crystal rocking curve, the spectrum generated by rocking the crystal will be that of the source broadened by the crystal. The shape of a peak in the spectrum will be that of the single crystal rocking curve. The loss of source intensity by using tighter collimation is overcome in the sub-kilovolt region by the fact the X-rays only need to be reflected off of one crystal. To measure the resolution of a curved crystal, instead of rocking the crystal, the detector with a slit in front of it is scanned.

A useful source at low energy X-rays is the broad nickel L_β line (7 eV wide at 868.8 eV) absorbed by the sharp K-edge of neon {1}. The neon photon absorption spectrum consists of a series of lines due to the excitation of the 1s electrons to the 3p, 4p, 5p, etc. orbitals of the excited atom followed by the ionization continuum absorption. The 1s-3p resonant absorption (866.9 eV) is relatively strong compared to the rest of the K-shell absorption, very narrow (0.2 eV), and separated from the nearest resonant line, 1s-4p by 1.8 eV and the continuum (870 eV and above) by 3 eV. The width of the 1s-3p absorption line is estimated to be 0.2 eV by Z extrapolation of the experimental widths {2} of the final K state of the absorption. The width of the initial LIII state is an order of magnitude narrower than the K state, therefore it does not contribute to the width of the absorption line (see figure 1).

When the resolution of the curved crystal is to be measured there are two competing needs for collimation. In order to image the whole nickel L_β line a large angular area of the crystal needs to be illuminated, but it needs to be illuminated by X-rays collimated tighter than the resolving power of the crystal. The equation which governs the ratio of θ_i, the angular part of the crystal illuminated, to θ_S, the angular divergence of the beam is,

$$R_\theta = \theta_S/\theta_i = R \cos\theta \,/\, (D_2 + D_1 d_2 /(d_1+d_2)) \qquad (1)$$

The variables are defined in figure 2a. The ratio R_θ is a minimum when $d_1=0$. This implies that

$$R_\theta > R \cos\theta \,/\, (D_2+D_1) \qquad \text{for all } d_1, d_2 \qquad (2)$$

This imposes a minimum requirement on the source to crystal distance,

$$D_1+D_2 > R \cos\theta \,/\, R_\theta \qquad (3)$$

if R and θ are fixed and R_θ must be less than a certain value.

The slit on the detector must be narrow enough to resolve the feature being examined. This implies that

$$s << D \tan\theta \, dE/E \qquad (4)$$

where dE is the energy width of the peak that needs to be resolved and E is the energy of that peak (see figure 2b). The angular size of the slit θ_d is

$$\theta_d \approx s/D \qquad (5)$$

In order to deconvolve the various broadening effects, assume that the response of the source collimation, θ_S, the source energy width,

ε_s, the crystal resolving power, θ_c, and the detector angular size, θ_d, (θ_d applicable on for curved crystal measurements) are all gaussian curves convolved together to yield the final response function. Using this assumption the width of the peak is

$$W = \mathrm{sqrt}(K^2(\theta_s{}^2 + \theta_c{}^2 + \theta_d{}^2) + \varepsilon_s{}^2) \qquad (6)$$

where

$$K = E \cot\theta, \qquad (7)$$

E is the mean energy of the X-rays and θ is the bragg angle for X-rays of energy E. Once W is measured, equation 6 can be manipulated to yield a value for the energy resolution of the crystal,

$$\varepsilon_c = K\,\theta_c = \mathrm{sqrt}(W^2 - K^2(\theta_s{}^2 + \theta_d{}^2) - \varepsilon_s{}^2) \qquad (8)$$

It should be noted that if

$$\varepsilon_c{}^2 << K^2(\theta_s{}^2 + \theta_d{}^2) + \varepsilon_s{}^2 \qquad (9)$$

the sensitivity is

$$S = (d(\varepsilon_c)/dW)/(\varepsilon_c/w) = (W/\varepsilon_c)^2 >> 1 \qquad (10)$$

making it difficult to obtain an accurate calculation of ε_c from a measurement of W. The opposite is true if

$$\varepsilon_c{}^2 >> K^2(\theta_s{}^2 + \theta_d{}^2) + \varepsilon_s{}^2 \qquad (11)$$

In this case $S \approx 1$, and $\varepsilon_c \approx W$.

EXPERIMENTAL

The single crystal rocking curve for the flat mica crystal was measured using the setup shown in figure 3a. Muscovite mica cleaved along the 002 plane to a thickness of 0.13 mm and cut in a square 3.8 by 3.8 cm was used. The thin sheet of mica is optically contacted to a glass flat so that the deviation in the angle of its surface is less than 3×10^{-4} rad. A Henke source with a solid nickel anode was used to generate the X-rays. The anode potential was 2.5 KV with a current of 60 mA. The system was maintained at a pressure of 5×10^{-6} torr. The angular divergence of the source in the horizontal direction, θ_S, was 8×10^{-4} rad and 20 mrad in the vertical direction. A gas proportional counter with P-10 gas maintained at a pressure of 350 torr was used to detect the X-rays. The gas cell is 5.1 cm long with polypropylene windows reinforced with nickel grid (200 lp/mm, 70% open area) on both ends. The reflected intensity as a function of angle with < 20 mtorr of gas in the cell and 12 ± 1 torr were measured. The two angular spectra are then reduced to yield the total scattering cross section of the neon gas as a function of energy. The FWHM of the 1s-3p absorption peak is measured. Using the known source collimation and the natural width of the absorption peak, the energy resolution of the crystal, ε_C, is calculated using equation 8. The term in equation 8 due to the slit in front of the detector, θ_d, is not applicable to this measurement.

The resolution of a 0.13 mm thick mica crystal curved to a 5.1 cm radius of curvature by clamping it between two metal forms was measured using the setup in figure 3b. The source collimation, θ_S, was 8×10^{-4} rad, and the detector collimation, θ_d, was 4.5×10^{-4} rad. 6.2×10^{-3} rad of the crystal were illuminated by the X-ray beam. The detector was set at the correct angle to measure the center of the L_β peak. The curved crystal was then rotated to maximize the count rate, thus orienting the crystal so the beam would subtend the correct range of angles for the L_β line. The detector was then rotated to measure the energy spectrum. This was done for neon gas cell at pressures of < 20 mtorr and 12 ± 1 torr. The data is reduced the same way as the data for the flat crystal except the term in equation 8 for the detector collimation must be used.

RESULTS

The angular spectra for the nickel L_β line with and without neon gas in the cell appear in figure 4. The two spectra were reduced to yield the neon photo absorption cross-section as a function of energy which is shown in figure 5. The FWHM of the 1s-3p absorption peak at 867 eV is 1.0 ± 0.1 eV. Using this number to calculate the crystal resolution yields, $\varepsilon_C = 0.72 \pm 0.15$ eV. The crystal curve calculated with the program ROCKIT {3} is shown in figure 5 as the curve labeled with M. The amplitude of the calculated curve has been modified to match that of the measured absorption peak. The FWHM of the calculated peak is 0.52 eV.

When the measurements were made for the curved mica crystal signal was detected (0.5 counts/sec) but the count rate was too low to obtain a statistically significant answer in a reasonable amount of time.

CONCLUSIONS

The measured crystal resolution is 40% higher than the calculated value but it is consistent with the double crystal value measured at 930 eV {4}.

energy	measured single crystal resolution (method)	calculated resolution
870 eV	0.72 ± 0.14 eV (single)	0.52 eV
930 eV	0.72 ± 0.10 eV (double deconvolved)	0.52 eV

Although no meaningful data was collected for the curved mica crystal, if the source intensity can be increased by a factor of 4 to 8 (making the nickel anode as bright as the copper anode), a direct measurement of the resolving power of the curved mica crystal can be made.

REFERENCES

1. Liefeld, R.J.: Appl. Phys. Lett. **7**,276 (1965).
2. Parratt, L. G.: Revs. Modern Phys. **31**, 630 (1959).
3. Glinsky, M. E.: ROCKIT User's Guide (Version 1.1), RP-85-105, 1985.
4. Glinsky, M. E., Waide, P. A.: Resolving Power of Muscovite Mica (002), RP-85-91, 1985.

Figure 1.

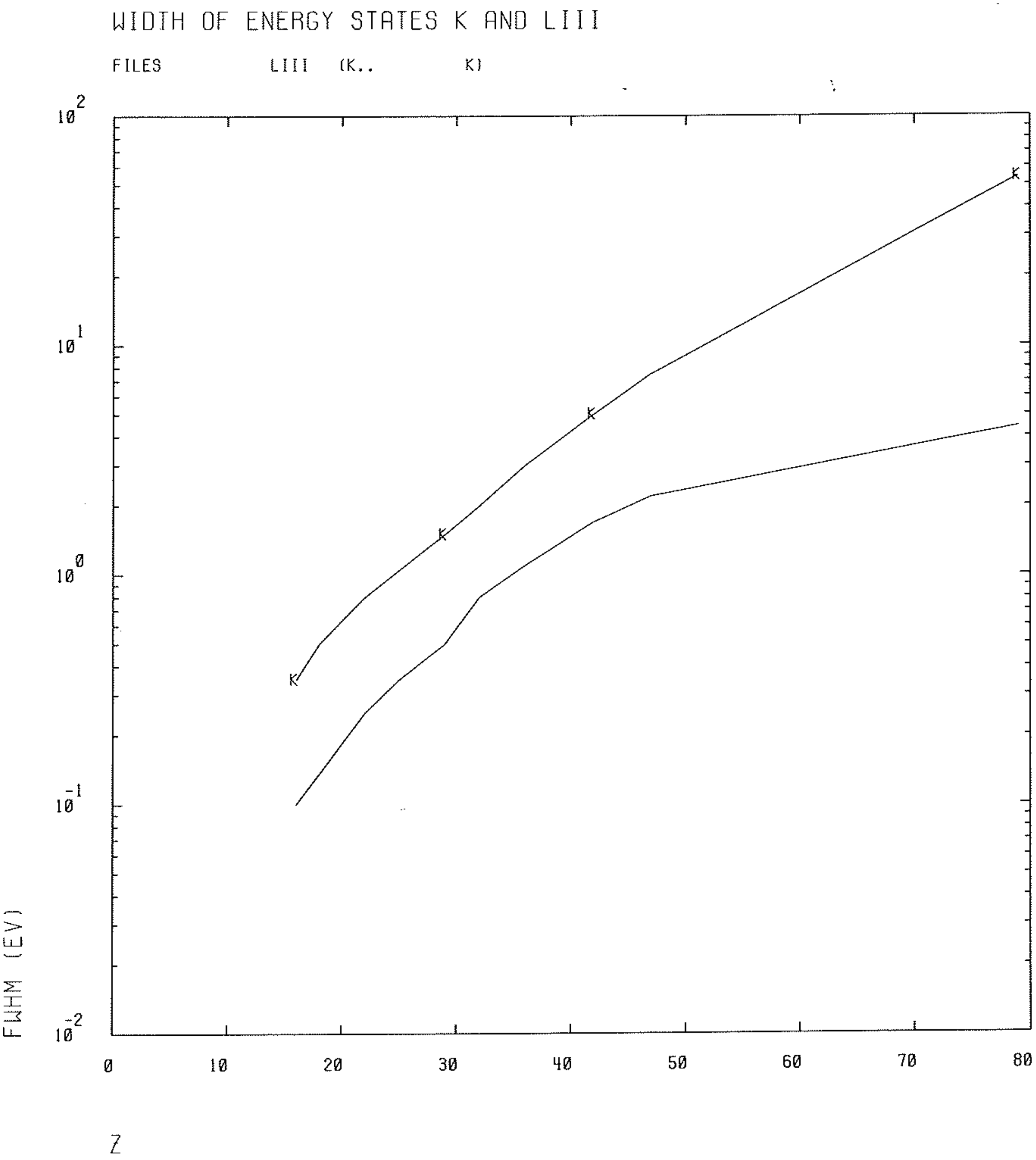
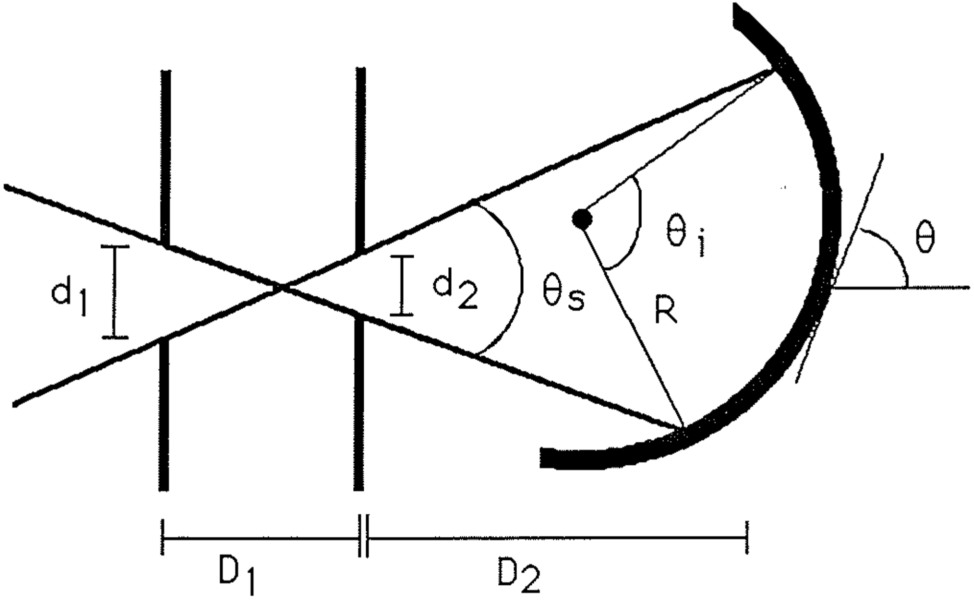

WIDTH OF ENERGY STATES K AND LIII
FILES LIII (K.. K)
10^2
10^1
10^0
10^-1
10^-2
FWHM (EV)
K
K
K
K
K
0
10
20
30
40
50
60
70
80
Z

Figure 2a.



$$\theta_s = \frac{d_1 + d_2}{D_1} \qquad \theta_i = \frac{d_2 + \theta_s D_2}{R \cos\theta}$$

Figure 2b.

$$s = -D \tan\theta \, dE/E$$

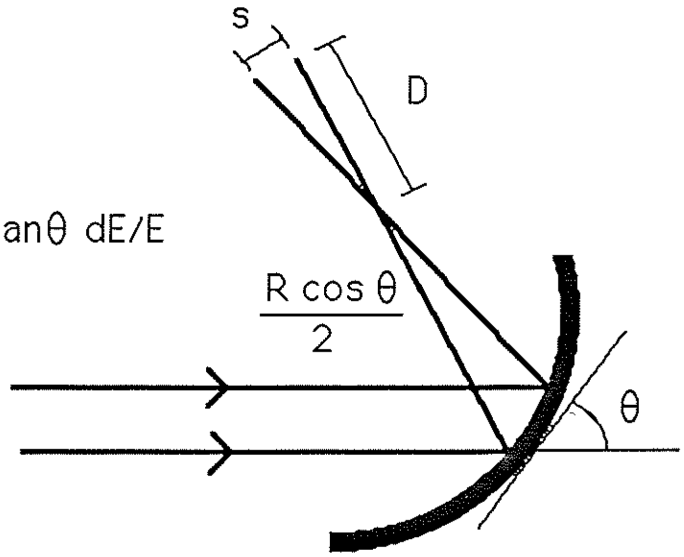


Figure 3a.

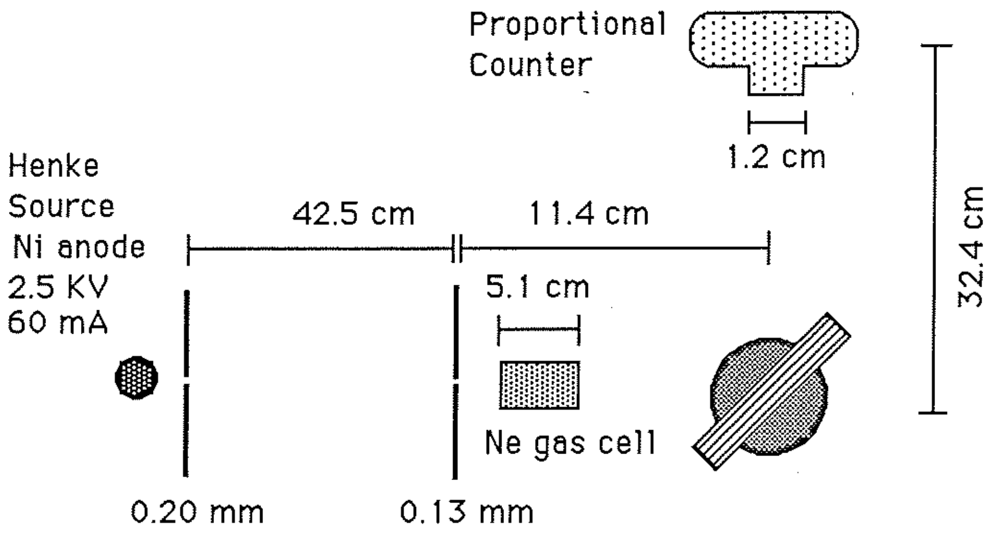


Figure 3b.

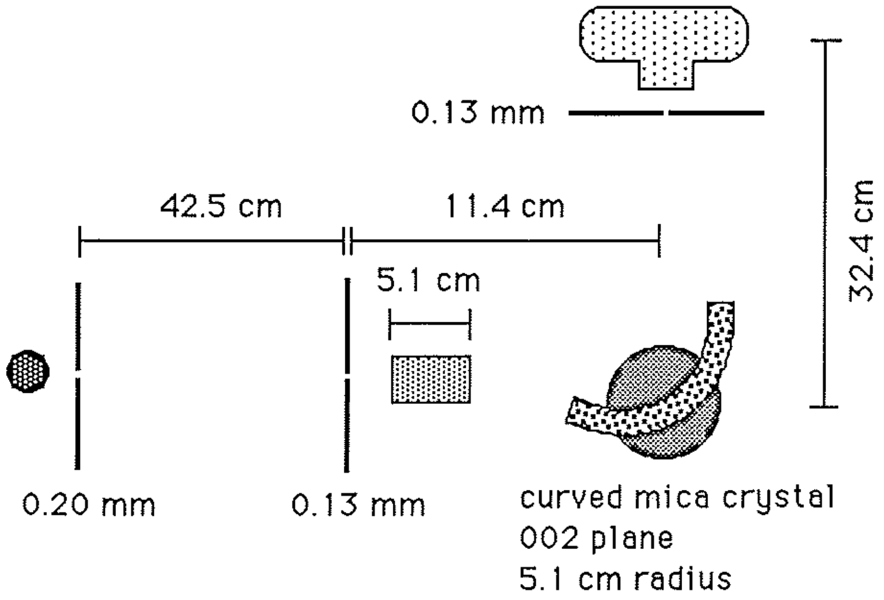


Figure 4.

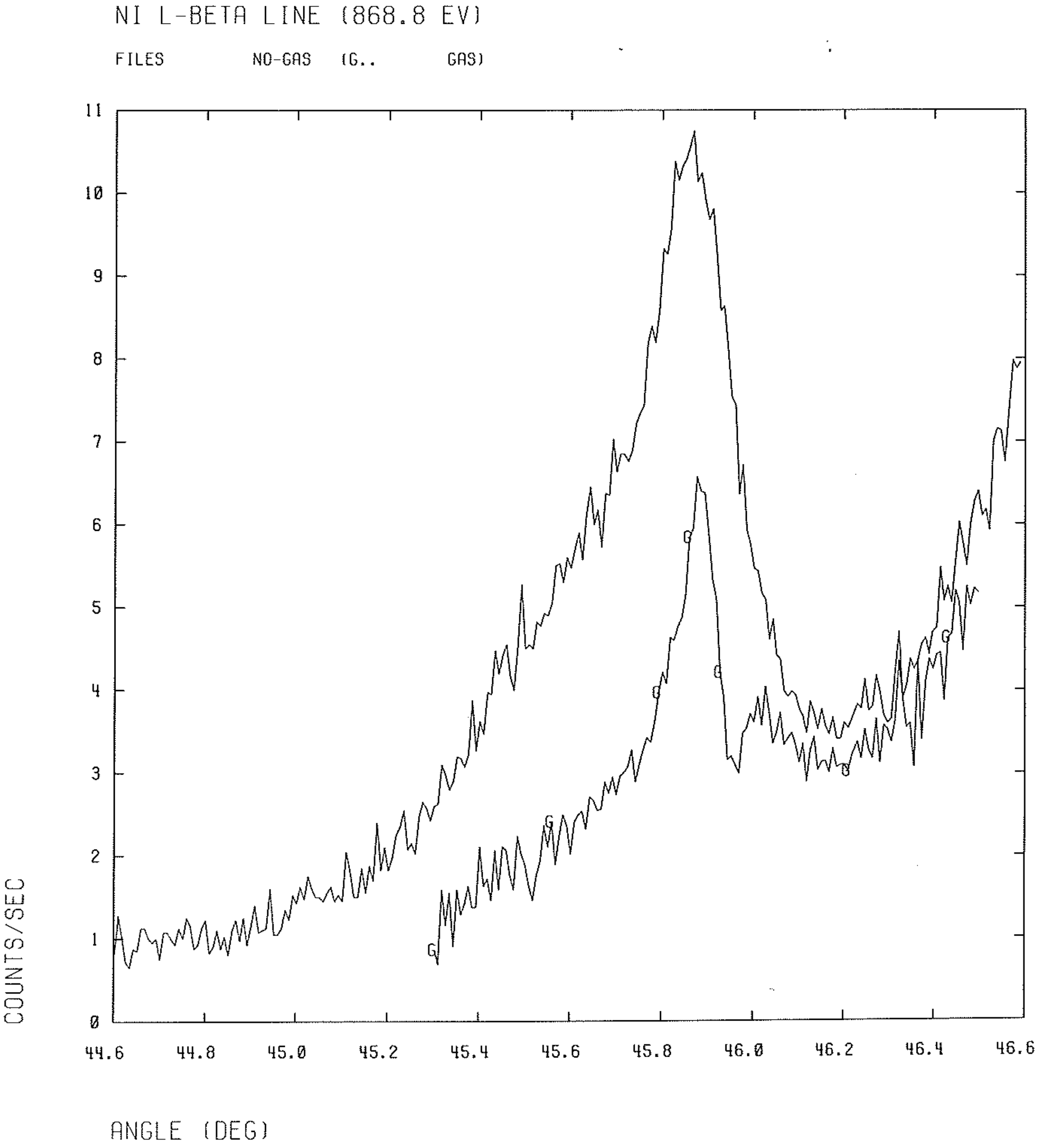
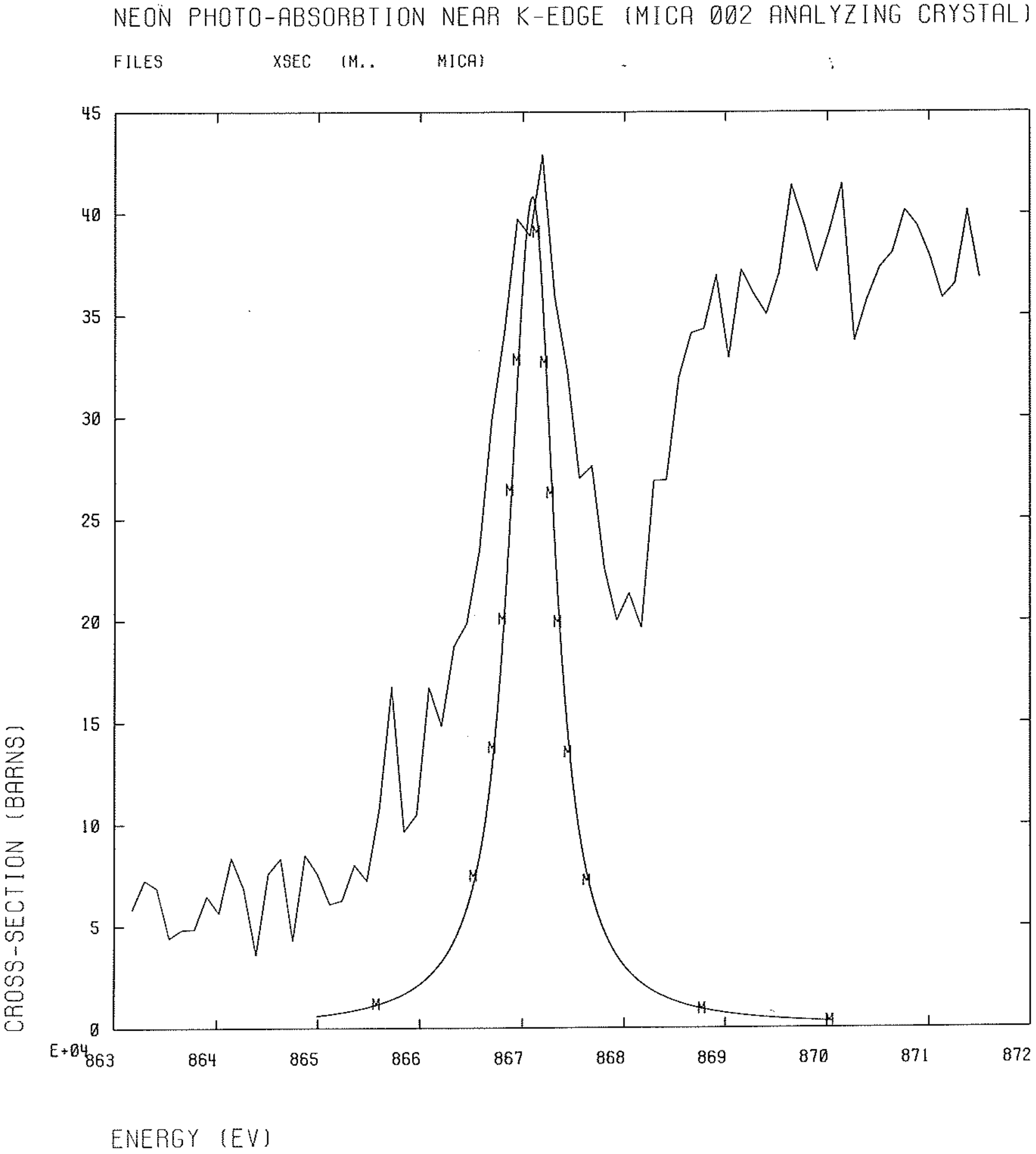

NI L-BETA LINE (868.8 EV)
FILES NO-GAS (G.. GAS)
COUNTS/SEC
0
1
2
3
4
5
6
7
8
9
10
11
44.6
44.8
45.0
45.2
45.4
45.6
45.8
46.0
46.2
46.4
46.6
ANGLE (DEG)
G

Figure 5.


NEON PHOTO-ABSORBTION NEAR K-EDGE (MICA 002 ANALYZING CRYSTAL)
FILES XSEC (M.. MICA)
CROSS-SECTION (BARNS)
0
5
10
15
20
25
30
35
40
45
E+04
863
864
865
866
867
868
869
870
871
872
M
ENERGY (EV)