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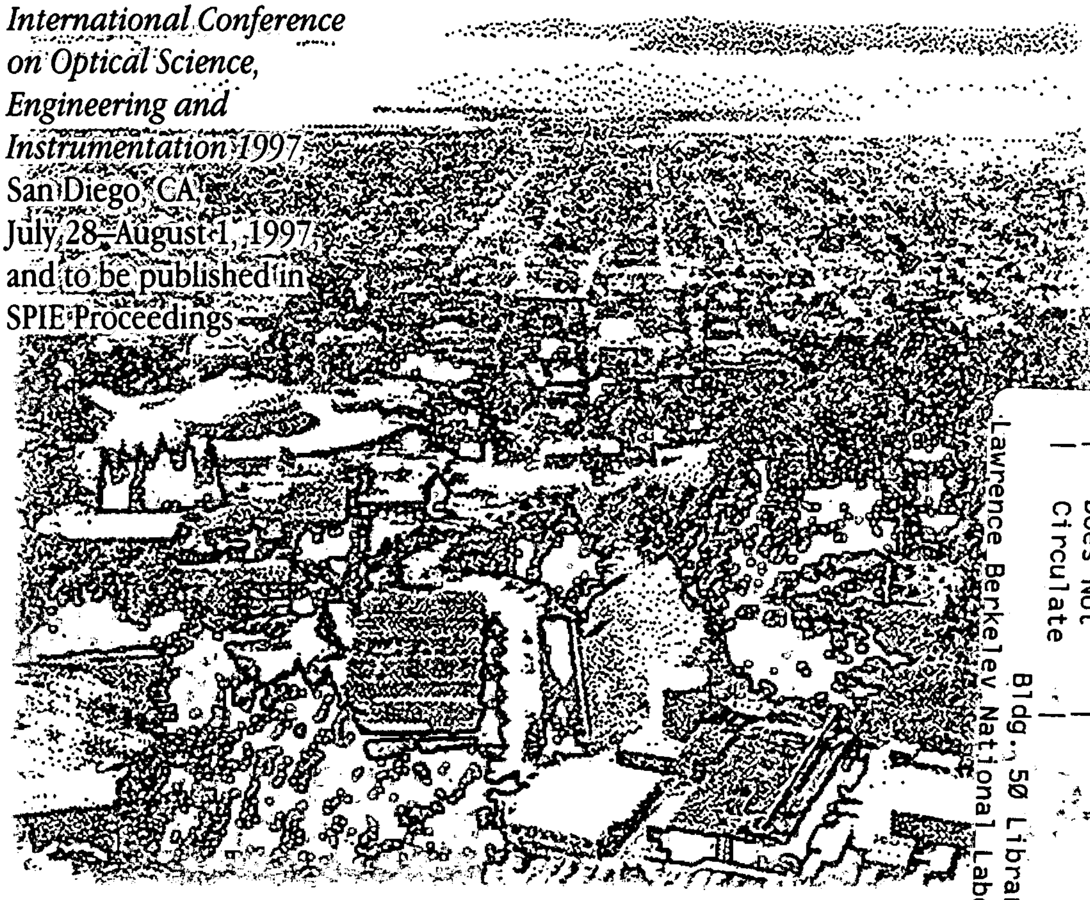

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ABSTRACT

We report here the development of a hard x-ray multilayer grating that has achieved an absolute efficiency of 34% at a wavelength of 1.54Å. The W-C multilayer itself has a reflectivity of 57% and the grating has a 0th order absolute efficiency of 36%. The origin of this extraordinarily high efficiency is that the short period and highly asymmetric structure of the grating combined with its deep grooves allows light to interact with a large number of layer pairs. This increases angular separation of the diffraction orders and reduces the multilayer bandwidth to the point where there is little or no order to order overlap in the grating structure, and hence maximum intensity can be diffracted into a selected order. This paper reports on the development of an optimized multilayer grating and some of its unique characteristics.

Keywords: multilayer gratings, diffraction gratings, multilayer mirrors, x-rays

INTRODUCTION

The properties of multilayer gratings (MG) have attracted much interest over decade due to their possible application in many types of X-ray optical systems. They can combine both the dispersion and high resolving power of the grating with the high reflectivity at non-grazing incidence angles given by multilayer mirrors. Pioneering work of Barbee[1] and the later work of Warburton[2] showed that by choosing the correct multilayer period and grating period it is possible to preferentially diffract light into a selected grating order, thus maximizing the diffraction efficiency and giving the possibility of working at high resolution by the use of higher orders.

Even though scalar models have been developed in order to explain the behavior of multilayer gratings, precise modeling has not been possible due to the absence of a rigorous theory. The scalar theories can not answer the questions: regarding maximization of diffraction efficiency, orders be supressed, how does the period, groove depth and groove width affect the diffraction efficiency. The accurate study of MGs became available after two rigorous theories, the "differential method" [3,4,5], and "modal theory" [6,7] were developed independently in Marseille University (France) by Neviere and collaborators and in IPMT (Chernogolovka, Russia) by Martynov and collaborators. We compared these theories, and for the wide range of parameters examined they gave identical results [6].

During this work we discovered a new type of structure that strongly enhanced the intensity in a selected order, and we also experimentally confirmed this effect [6,7,8,9]. The key difference with earlier MGs was that these structures had a short period and deep grooves, and so a significant number of multilayer periods could be involved with the diffraction process, increasing efficiency in a selected order. For example in [8], we found experimentally that one structure gave an absolute -1 order efficiency of near 20%, compared to a zero order efficiency of 50%.

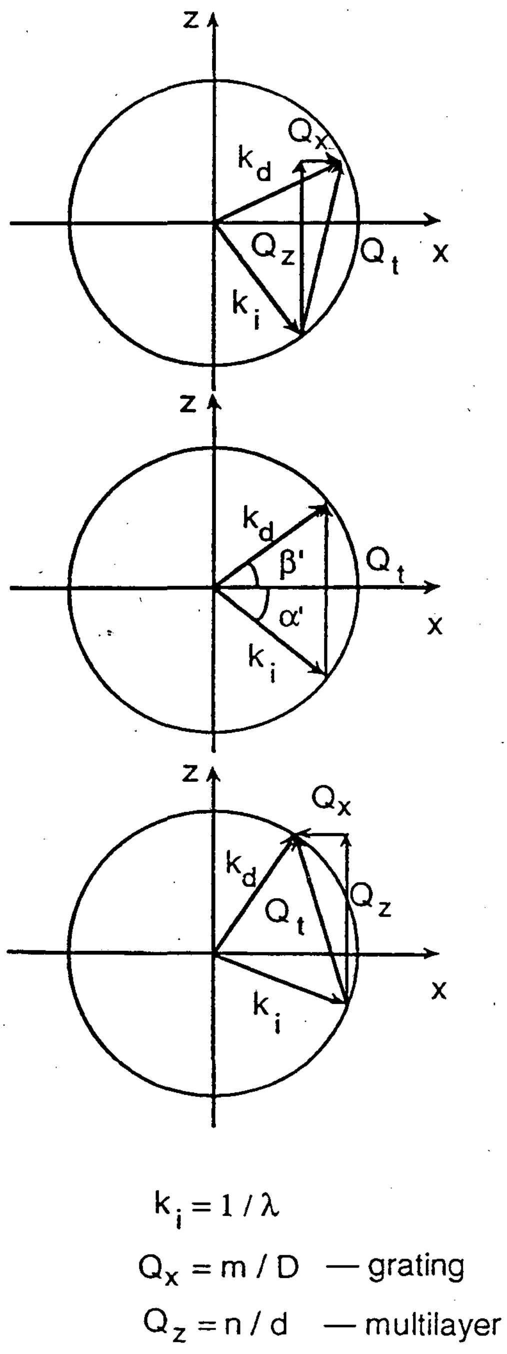

Fig.1 The reciprocal space diagram for -1, 0 and +1 orders. The radius of the circle is $1/\lambda$ and the grazing angles of incidence and diffraction are α' and β' respectively.

However, a land to groove ratio of 1:1 was used, resulting in strong 1st order diffraction, but weak higher orders. In this work, we give new results which report on an optimized MG structure in which the blaze effect is enhanced by making the land to groove ratio of the grating highly asymmetric, thus increasing the effective extinction depth of the multilayer. This reduces further the overlap of the multilayer diffraction with more than one grating order, and hence enhances efficiency in a selected order.

One of the key measurements we wished to perform was on the purity of the diffracted light at incidence angles corresponding to the maxima of particular orders. These maxima are best understood from a reciprocal space diagram. Fig. 1 shows the reciprocal space diagram for -1, 0 and +1 orders. The radius of the circle is $1/\lambda$ and the grazing angles of incidence and diffraction are α' and β' respectively, with the perpendicular momentum transfer of the multilayer given by $Q_z=1/d$ and for the grating by $Q_x=1/D$. d and D are the multilayer and grating periods respectively. Q_t is the combined momentum transfer. We can therefore see that the angle turned from the symmetric case is ϕ=md/nD. As the symmetric case is simply the Bragg condition, we therefore have $\alpha'=\theta-\phi$ and $\beta'=\theta+\phi$ where $\sin(\theta)=n\lambda/2d$. In this case with 1.54Å wavelength, a multilayer period of 45Å, and a grating period of 4µm, we find that α'=0.891°, β'=1.019° and ϕ=0.065° for +1 order. Grating orders are of course symmetric and spaced at equal values of ϕ.

The reciprocal space diagram also allows us to see the effect of a finite number of diffracting elements. The uncertainty in Q_z is simply due to the finite number of periods, i.e. $\Delta Q_z=1/dN^*$ where N^* is defined by the number of period in 1 effective extinction depth. The effective number of layers in an extinction depth has to be calculated from dynamical theory, but you can set bounds on the number from simple geometrical considerations. In the case of a shallow grating, where the number of grooves in one groove depth is much smaller than the extinction depth of a simple multilayer with the same parameters, the effective number of layers is just that in one groove depth (Fig. 2). As this number typically will be small (e.g. for a multilayer deposited on an ion etched grating structure), the angular width of the multilayer reflection will overlap many grating orders, thus reducing the light diffracted into the desired order. Clearly under these conditions, most of the light will be diffracted into zero order.

Of coarse, one can shorten the period to have the orders separated. This type of effect was described in details in refs. [6,8,9]. For a grating with very deep grooves, if one land has strong enough scattering that the transmitted intensity is small, the effective number of layers is that penetrated within the land. As an example, if we apply the conditions of this multilayer grating in zero order, the number of periods per land if it had a 1:1 land to groove ratio would be 8, and so the angular width of the reflection would be 0.13°. Note that this is twice the grating order to order separation, and hence we can expect to diffract light into several orders at once. To avoid this, in this case the lands were made thinner than the grooves, with a ratio of 1:4. In this way, rays can penetrate to almost twice the depth in the first groove. This would reduce the multilayer reflecting width to about the grating order to order value of 0.065°. However, the lands are thin enough that they will transmit a significant fraction of the radiation. The multilayer reflectivity starts to fall significantly at around 10 layer pairs. Thus the maximum path length in this case is around 5 μm. This has to be compared to the thickness of a single land in the optimized case described here of only 0.8 μm. Therefore, it is clear that several lands are involved in the diffraction process. Another way to avoid overlap of the multilayer diffraction with more than one grating order would be simply to make a weaker scattering multilayer. This could be done by using a lower Z heavy reflecting layer or by making the heavy layer thinner.

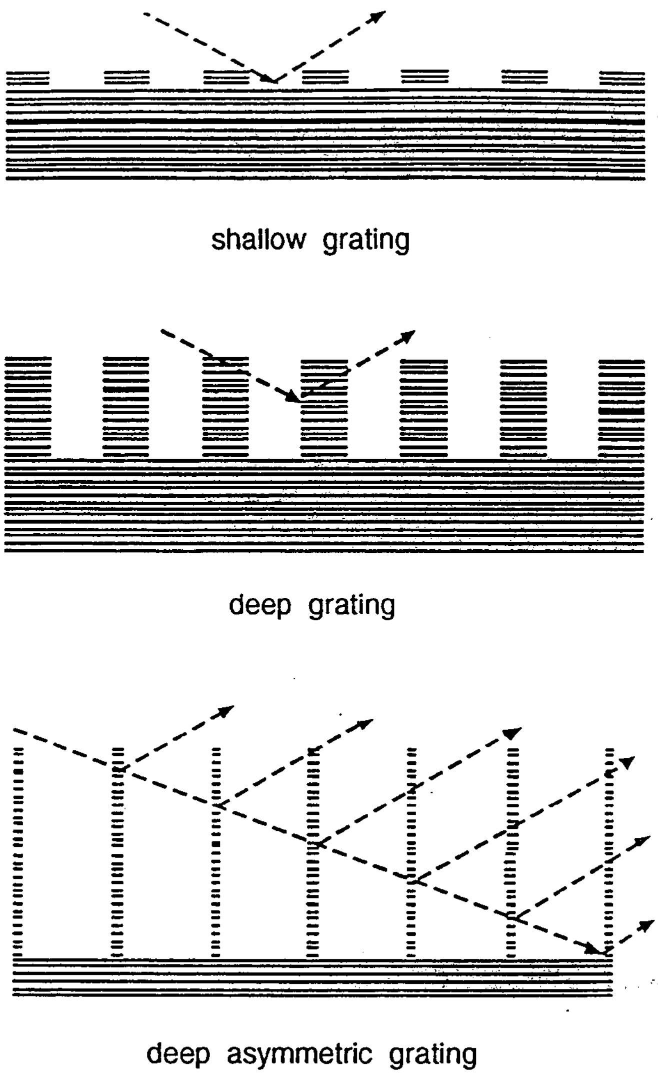

Fig.2 Different multilayer grating geometries showing the number of grating and multilayer periods involved in the one ray scattering process.

In this paper we describe the fabrication of an optimized grating, as well as experimental measurements of diffraction efficiency. The parameters of the multilayer grating were calculated with modal theory [Martynov et al, 6,7].

FABRICATION

The MG was fabricated in IPMT (Chernogolovka, Russia) by two methods. The first one consisted of four steps. First, the W/C multilayer mirror was deposited on a polished Si substrate. The period of the multilayer stack was 45Å, 30Å of C and 15Å of W. The magnetron sputtering machine SCM-651 designed for IPMT by B. Vidal (Marseille University, France) and manufactured by Alcatel was used for deposition. The multilayer coating consisted of 200 bilayers. This was necessary to optimize efficiency, but gave several technical problems that were overcame during the course of this work

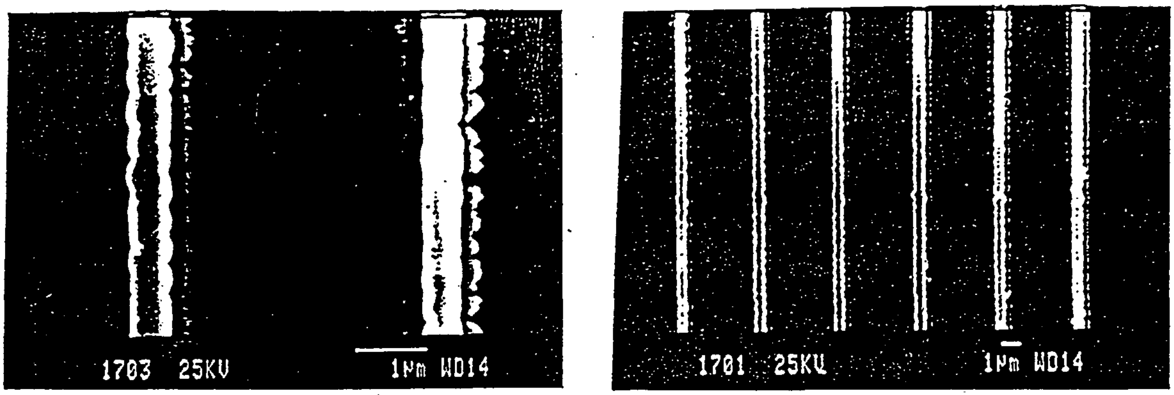

Fig. 3 Scanning Electron micrograph of a W/C multilayer grating on a Si substrate. Grating period is 4 μm, land/period ratio is 1:5, multilayer period is 45 Å (30 Å of C and 15Å of W)

The second step was production of a mask for photolithography. A grating pattern was formed in a layer of electron resist with an electron-beam lithography machine ZRM-12. The grating was of 4 μm period and $\approx 1{:}5$ land:period ratio. The width of the grating (perpendicular to the grooves) was 10 mm, and the length (parallel to the grooves) was 0.5 mm. (Fig.3) A wide grating was required due to the small Bragg angle of the multilayer mirror at the measurement wavelength of 1.54Å. After e-beam exposure the resist was developed and baked.

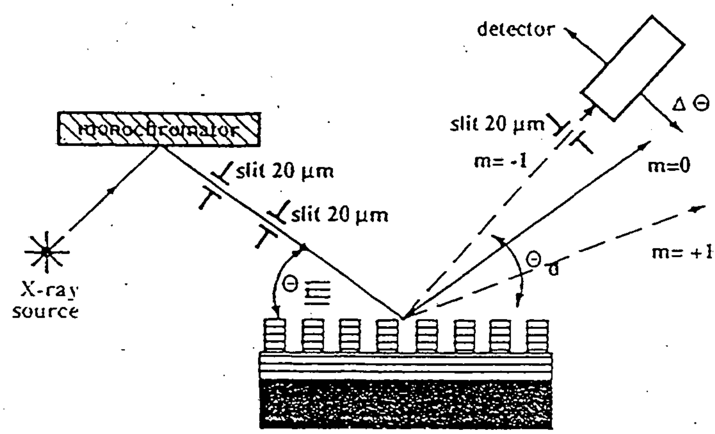

Fig. 4 Experimental X-ray setup for multilayer grating study. Θ_i is the incident angle, and Θ_d is the diffraction angle.

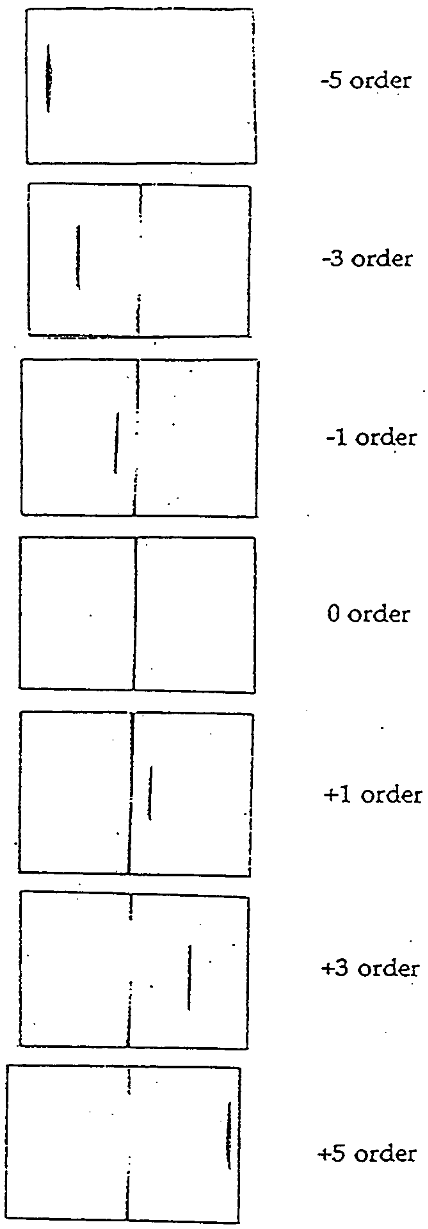

Fig. 5 Images recorded in the detector plane at incidence angles corresponding to the maxima of individual orders.

The third step involved photo-lithography exposure through the mask. The exposure time with the e-beam machine was rather long (about 2-3 hours)and expensive. This was the reason we decided to make a photo mask first and then use photo-lithography to produce a photoresist mask on the top of the multilayer mirror for the next step of ion-beam etching.

The last step was ion etching of the multilayer mirror through the photoresist mask formed in the previous step. The parameters of the ion beam were: Ar^+ ion energy - 850 eV, current density - 0.2–0.3 mA/cm^2, and the thickness of the photoresist was $1-1.2\mu m$. In order to obtain straight walls, the sample rotated around the incident beam axis at an angle of 70°. The absolute value of groove width error was $0.5\mu m$ due to the poor selectivity of the ion beam etching process. And therefore we could only etch to half of the necessary groove depth, since the required land width was about $1\mu m$

We therefore developed a new and more optimum method. The W/C multilayer mirror was deposited in the same manner. For e-beam lithography we used three layers instead of one. A $1.8\mu m$ thickness layer of photoresist covered the multilayer mirror surface, a $0.15\mu m$ layer of Al went on top of the first layer, and above the layer of Al a layer of e-resist of $0.3\mu m$ thickness was deposited. Even though the e-beam exposure was long and expensive we had to use it due to better resistance of e-resist to ion etching. We therefore made a grating pattern in e-resist with e-beam writing, developed and baked the resist. We then etched the Al and support photoresist with ions of Cl and O_2 through the e-resist mask. Finally we etched the multilayer mirror through the mask with ions of Ar and. O_2. The partial pressure of O_2 was 4–6x10^{-3} Pa. In this way we obtained deep grooves ($\approx 1\mu m$) with high dimensional accuracy.

MEASUREMENTS

The experimental study was carried out at the IPTM, and a schematic of the measurement arrangement is shown in Fig. 4 . A rotating anode x-ray source in combination with a Si(111) monochromator was used to produce a highly parallel and monochromatic beam. The beam was further collimated by a pair of 20 μm slits, and a detector also used the same slit size. The incident grazing angle and the angle of the detector with respect to the sample could both be scanned. In addition, a photographic plate could be located in front of the detector to simultaneously record all the diffraction peaks.

Fig. 5 shows a series of photographs recorded at a set of incident angles corresponding to maxima in the diffraction intensities for particular orders. Note that the grating was only etched into the central part of the mirror, so the multilayer reflectivity is visible at the upper and lower edges of each frame. It can be seen that in each frame, the central grating part of each image only has one order (except +1, where 0th order is visible as well).

All the orders are suppressed except one, for which the Bragg condition is satisfied, and hence the intensity in this particular order is maximized.

Fig. 6 shows a composite rocking curve for the multilayer grating made up of 15 individual rocking curves for -8 through + 6 order. In this case, the detector is put at a position that gives the maximum diffracted intensity for a particular order, and the grating is rocked over a small range. This is repeated for all orders, and then the intensities are added to give the composite curve shown in Fig. 6. The zero order diffraction efficiency was measured to be 36%, compared to the un-etched multilayer reflectivity of 57%. The -1st order is around 95% of the 0th order, with an absolute diffraction efficiency of 34%. It can also be seen that unlike earlier work in which symmetric gratings were used, it is now possible to diffract large intensities into high orders.

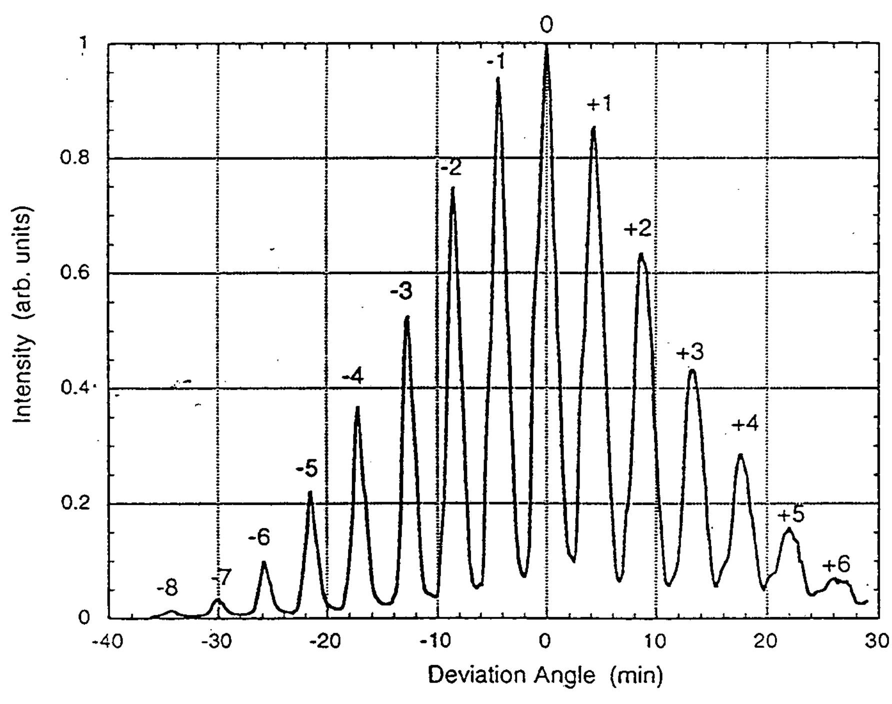

Fig. 6 Combined rocking curve of the multilayer grating.

CONCLUSION

This work has shown that it is possible to achieve very high diffraction efficiencies from multilayer gratings at hard x-ray energies. The high absolute diffraction efficiency, the absence of other orders when the incidence angle is set to maximize intensity in one particular order, and the large number of orders it is possible to diffract into with selection of the correct incidence angle, all result from the use of a deep groove structure and an asymmetric grating shape. This type of structure is designed to avoid overlap of the multilayer diffraction peak simultaneously with more than one grating diffraction peak.

Multilayer gratings can be expected to take an increasing role in high resolution monochromators and spectrographs. The demonstration that high diffracted efficiency can be achieved in a high order opens the way to their use as replacements for monochromator crystals in the difficult 1 - 2 KeV range. In addition, within the multilayer wavelength bandpass they are truly dispersive, and so could be the basis of high energy spectrographs of high efficiency.

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REFERENCES

1. T. W. Barbee, "Combined microstructure x-ray optics", Rev. Sci. Instr. 60(7), (1989), p 1588
2. W. K. Warburton, "On the properties of multilayer coated plane gratings", Nucl. Instr. Meth., A291, (1990), 278-285
3. M. Neviere, P. Vincent, and R. Petit, Nouv. Rev. Opt. 5, 65 (1974)
4. M. Neviere, "Bragg Fresnel multilayer gratings: electromagnetic theory". J. Opt. Soc. Am., A11, (1994), 1835-1845
5. M. Neviere & F. Montiel, Electromagnetic theory of multilayer gratings & zone plates", SPIE Vol 2805, (1996), 176-183
6. V. V. Martynov, B. Vidal et. al, "Comparison of modal and differential methods for multilayer gratings", Nucl. Instr. Meth. A 339, 617-625, (1994)
7. V. V. Aristov, A. I. Erko, and V. V. Martynov, "Dynamic x-ray diffraction by a multilayer mirror modulated with a transverse acoustic wave", Jour. of X-Ray Sc. and Tech., v. 3, 211-221, (1992)
8. A. I. Erko, B. Vidal, P. Vincent, Yu. A. Agafonov, V. V. Martynov, D. V. Roschupkin, M. Brunel, " Multilayer gratings efficiency: numerical and physical experiments", Nucl. Instr. Meth. A 333, 599-606, (1993)
9. A. Erko, V. Martynov, et. all, "Multilayer diffraction grating properties", Jour. Phys. III, France, v. 4, 1649-1658, (1994)

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