



**17<sup>th</sup> Asian Physics Olympiad**

**1-9 May 2016**

Experimental Problem – E1

# **Reflected Optical Diffraction Patterns from One-Dimensional Structures**

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# Our Discovery: self-assembled ZnSe nanograting

G.Wang,.....I.K SOU, Nanotechnology, 20, 215607 (2009)

**Sample: Fe/ZnSe bilayer**

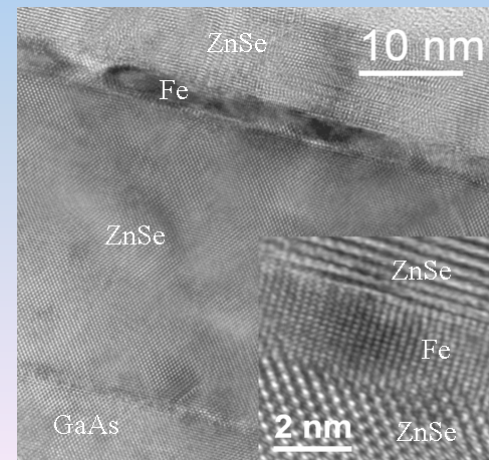
**Fe (2 nm)**

**ZnSe (30nm)**

**GaAs (100)**



The RHEED pattern taken right after the growth of an Fe layer on top of a ZnSe layer.



**Cross-sectional HRTEM image**



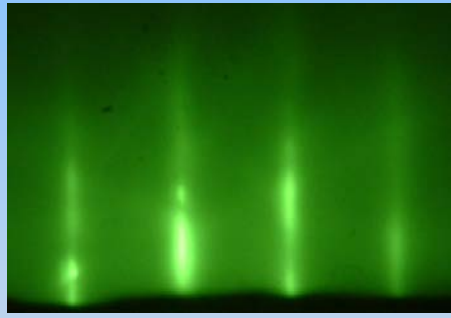
# RHEED observation of an annealed Fe/ZnSe bilayer

Fe (2nm)/ZnSe(30nm) bilayer annealed at 460°C for 10min

Standard RHEED Pattern for smooth thin films

0°

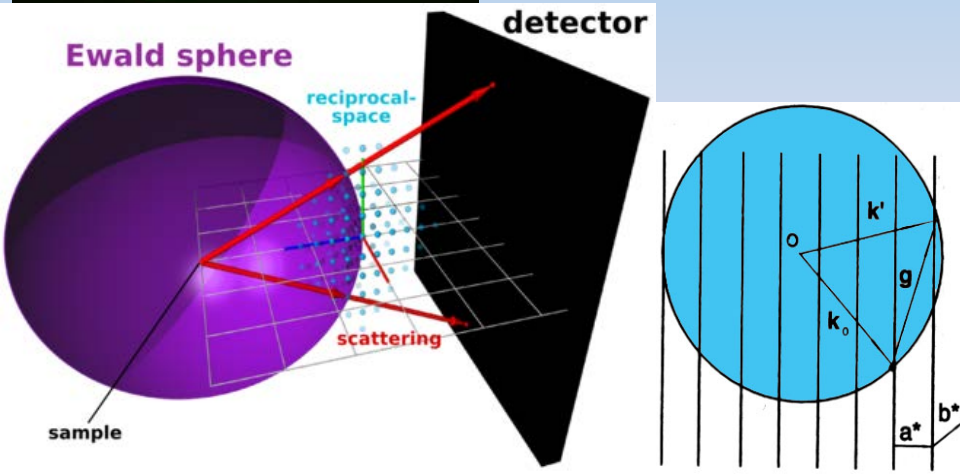
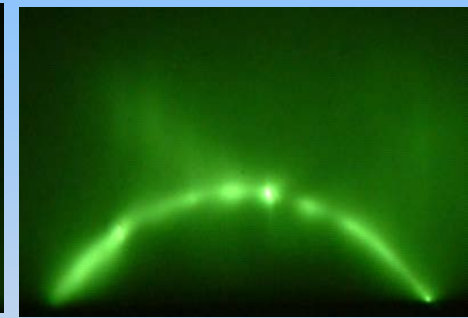
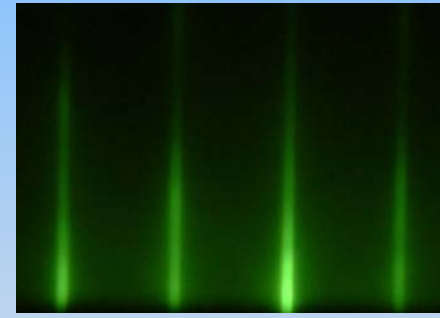
90°



Unusual RHEED observation

0°

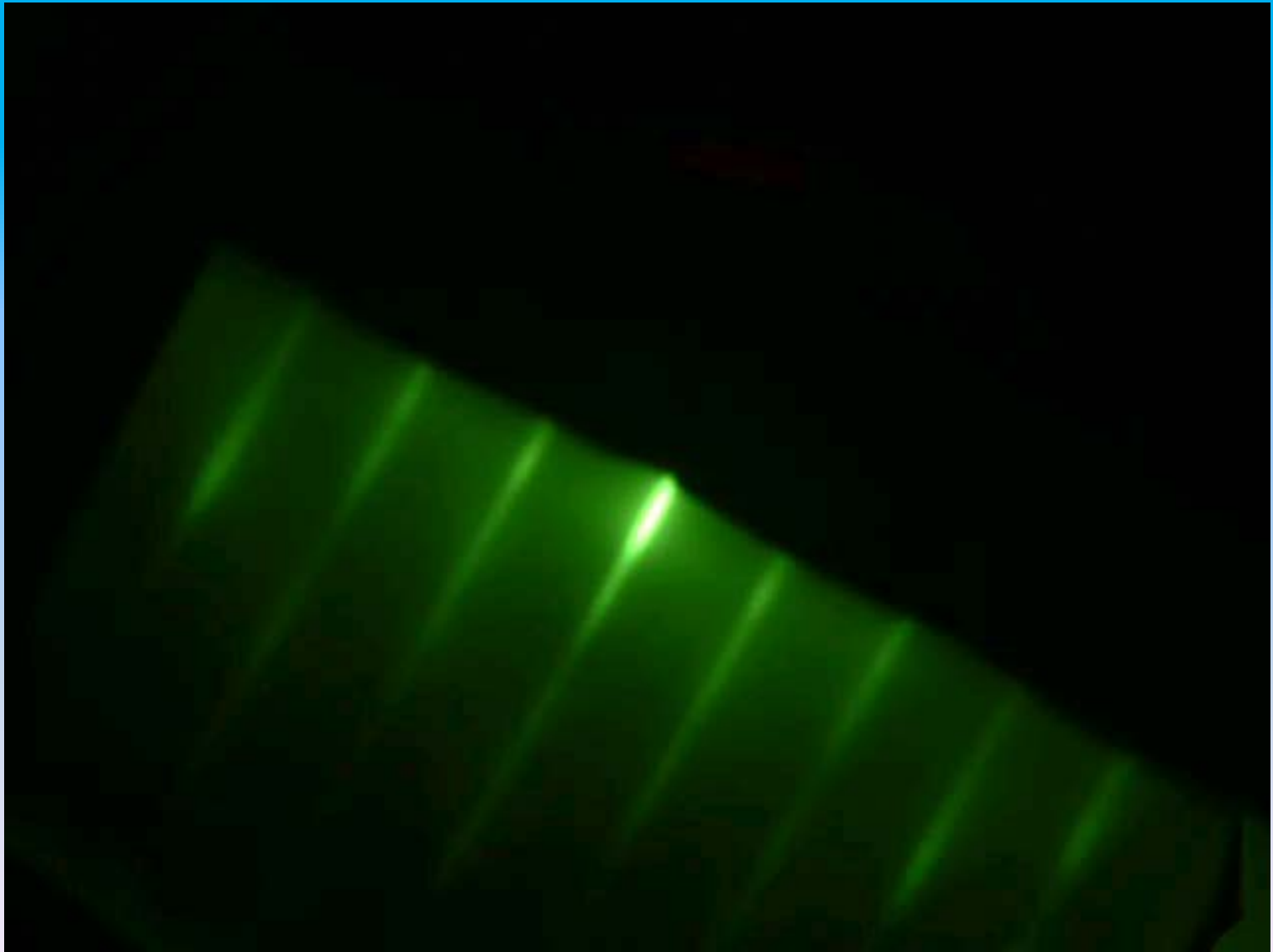
90°



What are the corresponding reciprocal lattice of this bilayer?



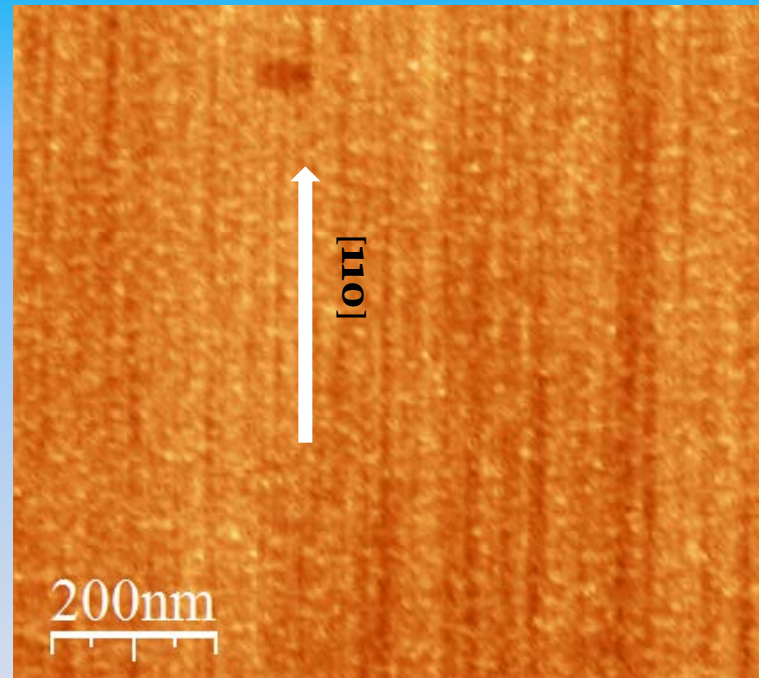
# RHEED Movie of the annealed Fe/ZnSe bilayer





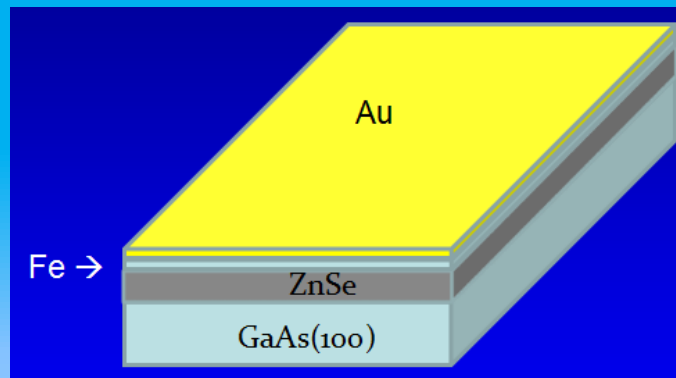
# Ex-situ AFM imaging

Its *ex-situ* AFM image clearly shows a 1D nanostructure aligned along the  $[110]$  direction though the resolution is poor due to air contamination.

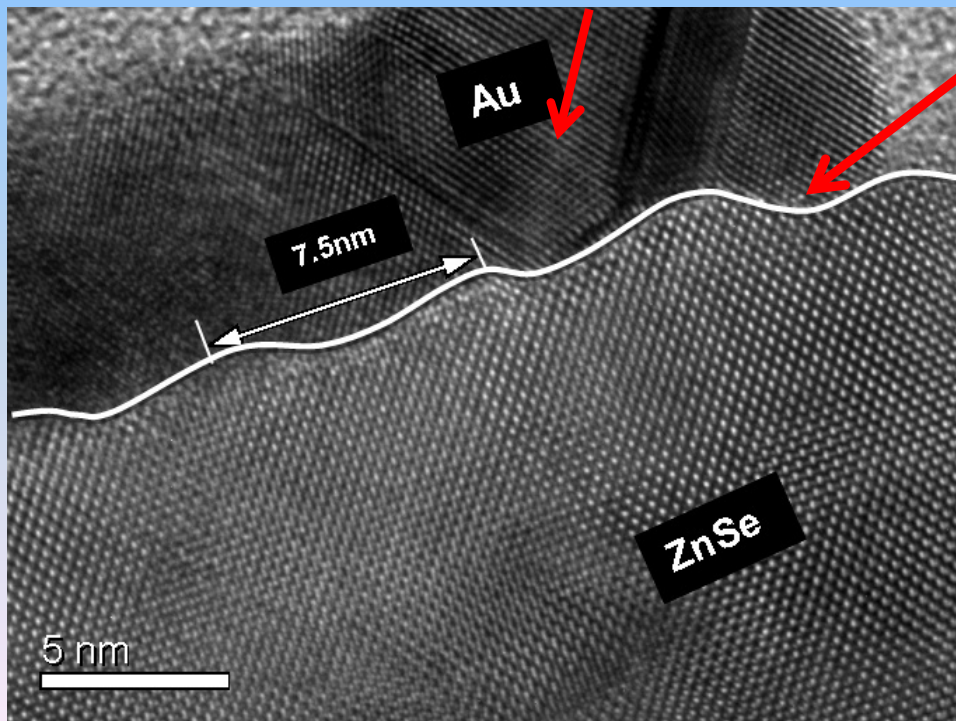




# HRTEM-cross-sectional image of an annealed Fe/ZnSe bilayer with a Au cap



Au film used to protect the surface



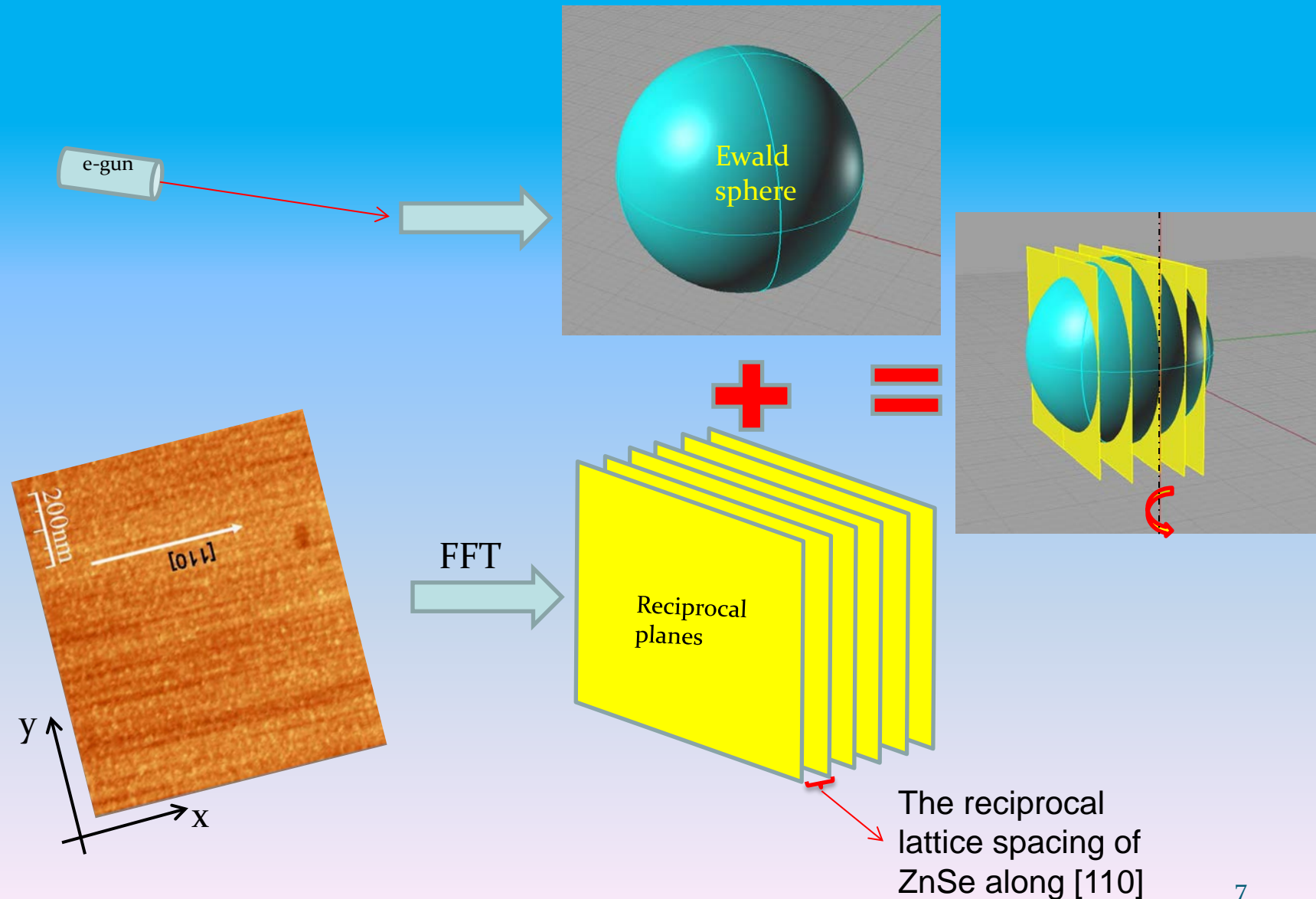
The wavy boundary between the gold layer and ZnSe layer is the cross section view of one-D structure array.

Apparently, the spacing of the 1D structures is 7 to 10 nm, while the height is about 1 to 2 nm.





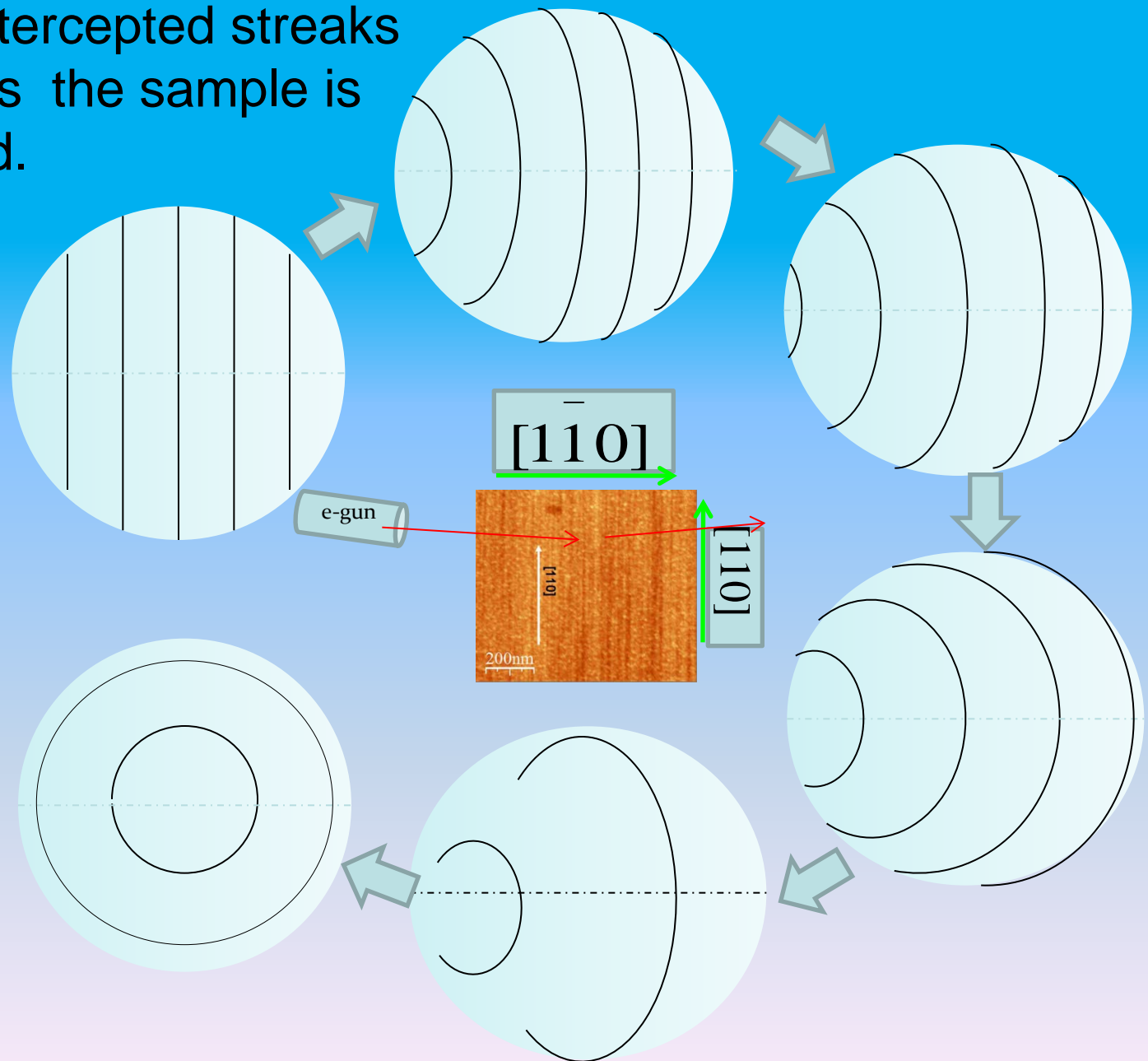
# Ewald sphere and Reciprocal lattice





The intercepted streaks bent as the sample is rotated.

After a rotation of  $90^\circ$







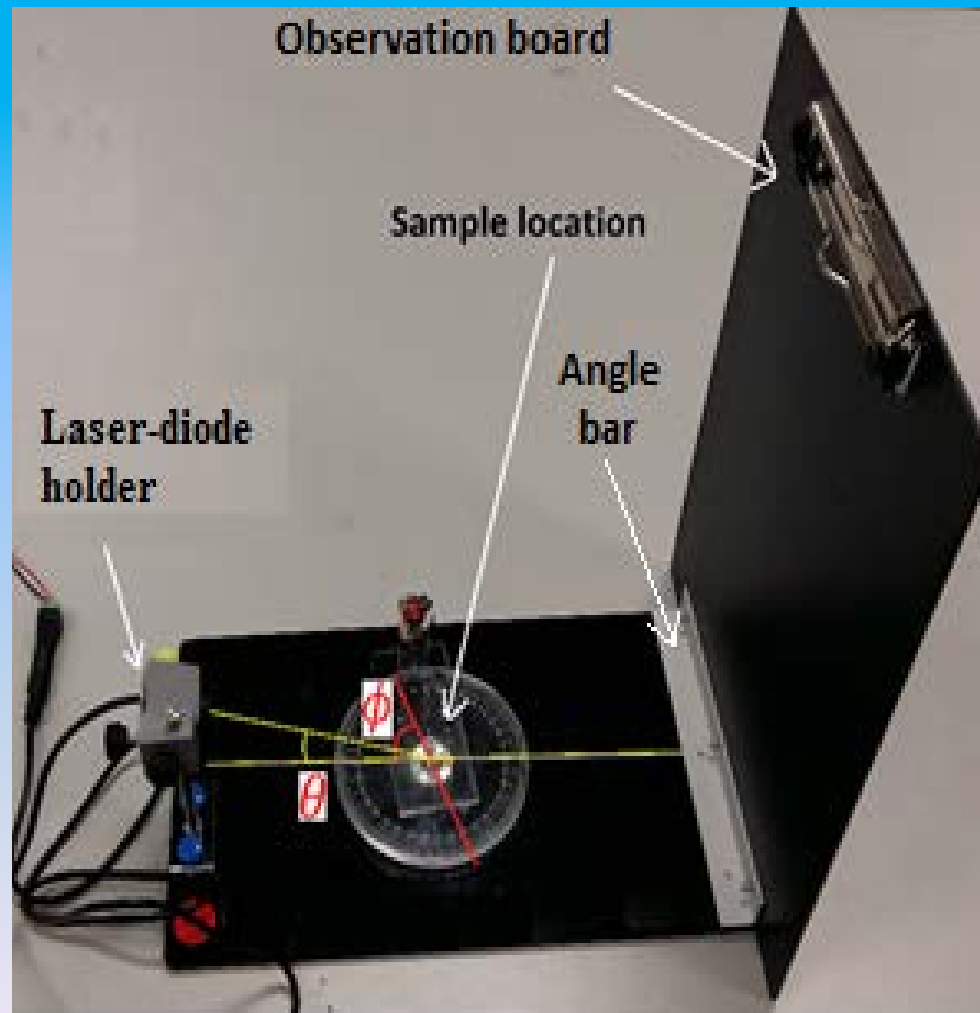
# ***Optical Analogy***

**Onsite Demo!**



## APhO Experimental Problem E1

# Reflected optical diffraction patterns from one-dimensional structures

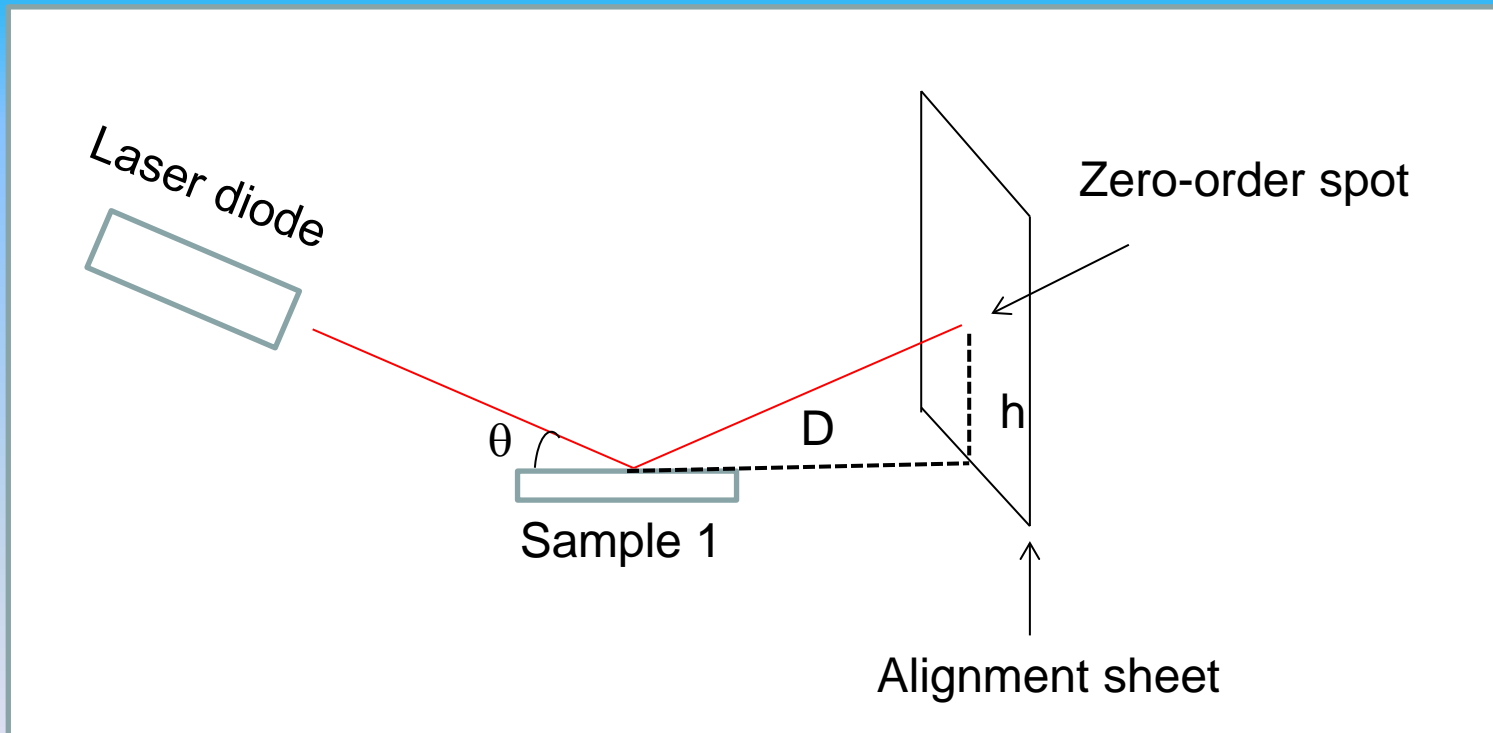


The setup used in Problem E1



# Part A: Alignment of the setup

## Sample 1: a plane mirror (Si wafer)

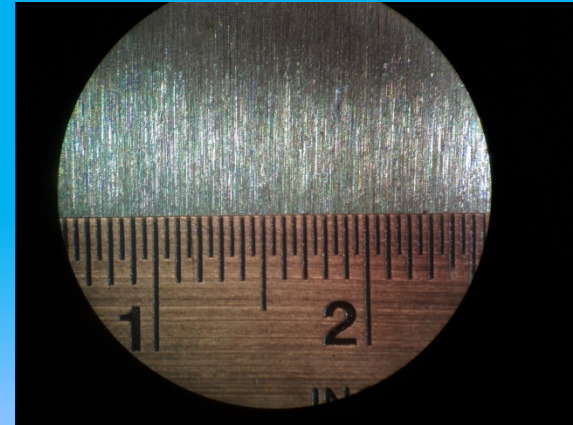


Students are asked to measure the height  $h$  (5.5 cm) and then find out the angle  $\theta$  ( $\sim 20.1^\circ$ )

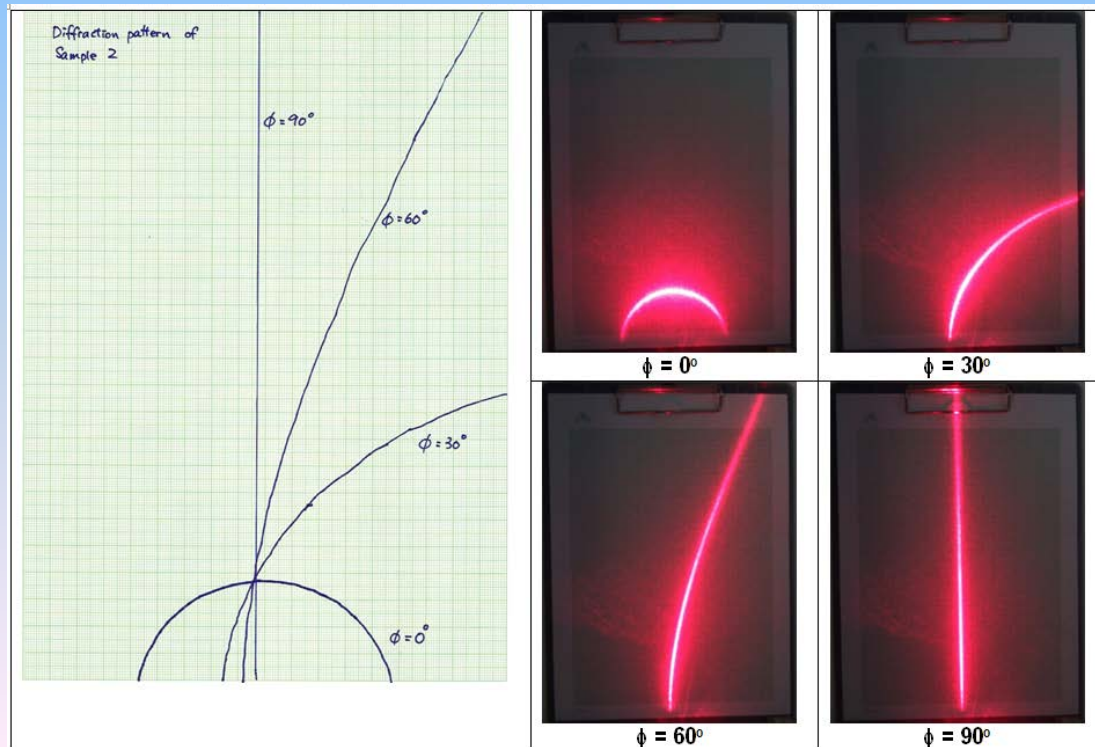


## Part B: Reflected Diffraction from Sample 2

**Sample 2: a steel plate with non-uniformly spaced scratch grooves**



**Task B1:** Students are asked to record diffraction patterns for four  $\phi$  values

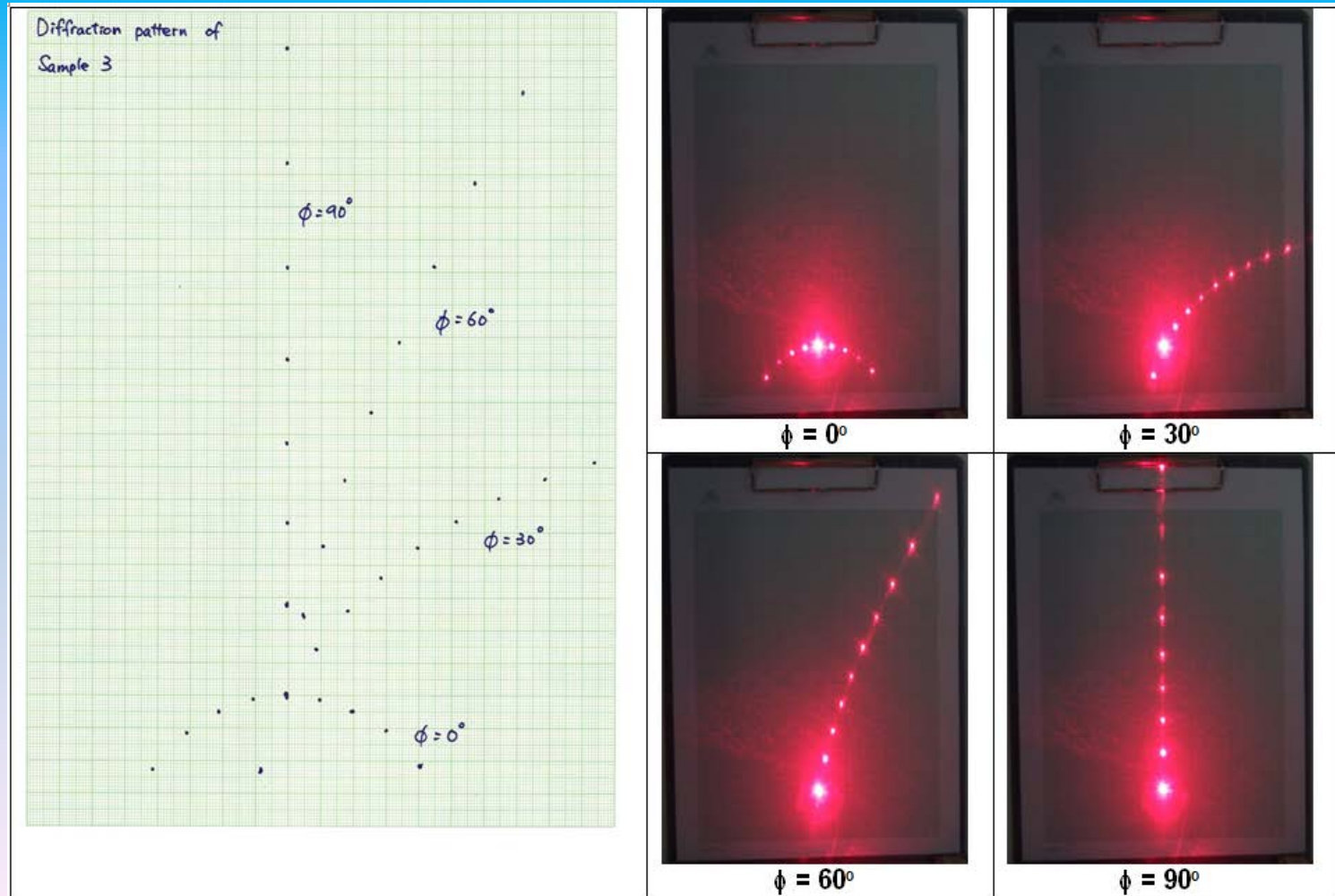




## Part C: Reflected Diffraction from a regular grating

**Sample 3: a regular grating with equal-spacing grooves  
( a Si wafer with periodic 1D photoresist pattern)**

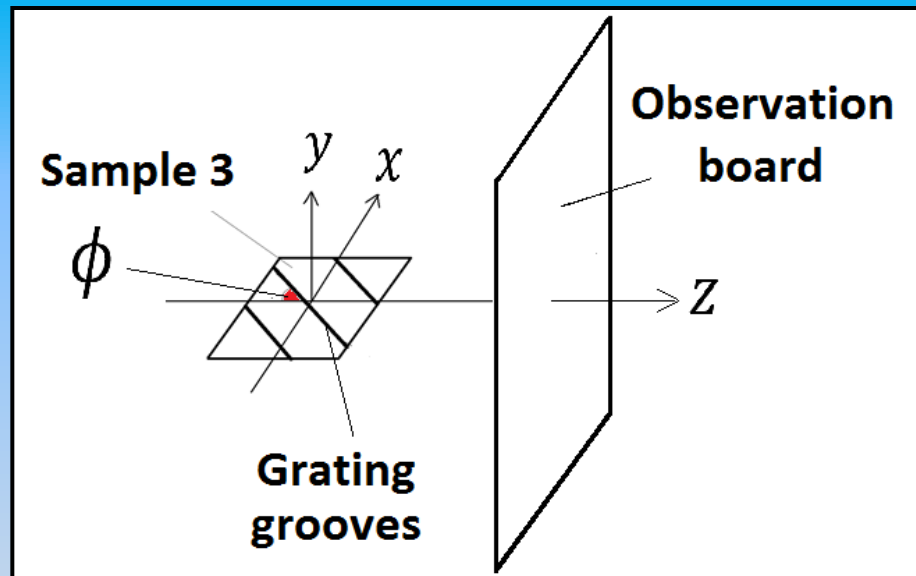
**Task C 1:** Students are asked to record diffraction patterns for four  $\phi$  values





## Part D: Theory behind the reflected diffraction patterns from Sample 3 (regular grating)

Using wave optics, (**credits go to Prof. Michael KY Wong**), one can derive the following two equations for the x and y coordinates of the diffraction spots:



$$y^2 = \frac{(D \cos \phi + x \sin \phi)^2}{\cos^2 \theta \cos^2 \phi} - x^2 - D^2 \quad (1)$$

$$x = \frac{D m \lambda \cos \phi}{a \cos \theta - m \lambda \sin \phi} \quad (2)$$

where “a” is the grating constant of Sample 3





# Tasks D1 (cancelled during the board meeting)

Tasks		Marks
D1	Based on Eqs. (1) and (2), the diffraction spots for $\phi = 90^\circ$ should lie along the y-axis at $x = 0$ . Derive an equation for the y values of the diffraction spots for $\phi = 90^\circ$ as a function of $a$ , $\theta$ , $\lambda$ , $m$ and $D$ . Write down your result in the answer sheet.	1.4

## Solution of D1 :

For  $\phi = 90^\circ$ , Eq. (2) gives the value of  $x$  equals to 0. To obtain the required equation, substitute Eq. (2) into the first term of Eq. (1). We have

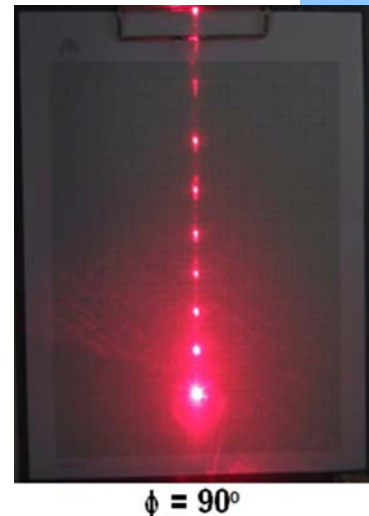
$$y^2 = \frac{\left( D \cos \phi + \frac{Dm\lambda \cos \phi}{(a \cos \theta - m\lambda)} \right)^2}{\cos^2 \theta \cos^2 \phi} - D^2$$

$$y^2 = \frac{(Da \cos \phi \cos \theta - Dm\lambda \cos \phi + Dm\lambda \cos \phi)^2}{\cos^2 \theta \cos^2 \phi (a \cos \theta - m\lambda)^2} - D^2$$

Cancellation of some terms leads to

$$y^2 = \frac{D^2 a^2}{(a \cos \theta - m\lambda)^2} - D^2$$

$$\therefore y = D \sqrt{\frac{a^2}{(a \cos \theta - m\lambda)^2} - 1}$$



## Tasks D2 - D4 (changed to D1 – D3 during the board meeting)

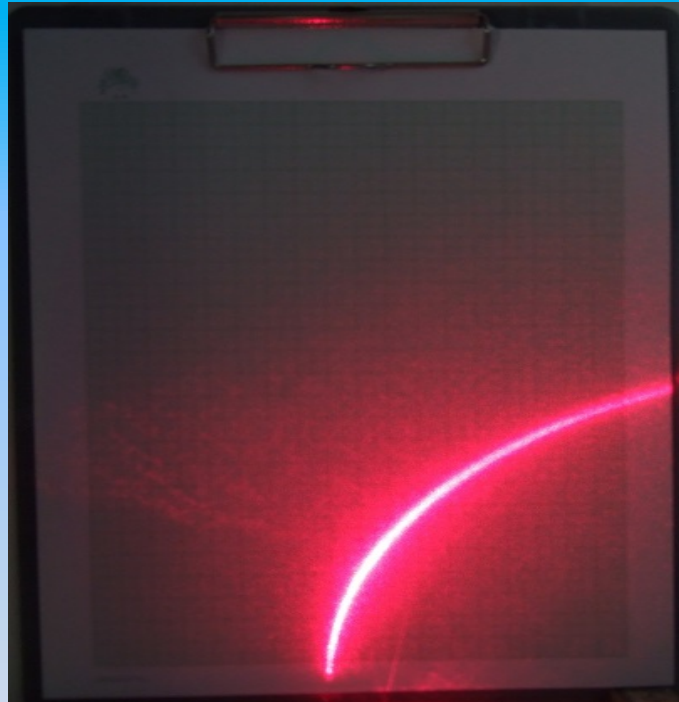
D2	<p>The solution for Task D1 can be rearranged to obtain a quadratic equation for the grating constant <math>a</math> of Sample 3, as</p> $Aa^2 + Ba + C = 0. \quad (3)$ <p>Derive the expressions for <math>A</math>, <math>B</math> and <math>C</math>. Enter your results in the corresponding table in the answer sheet.</p>	0.9
D3	<p>By solving this quadratic equation and using the measured <math>y</math> values of the diffraction spots for Sample 3 at <math>\phi = 90^\circ</math> (See Task C1), together with the known values of <math>D</math>, <math>\theta</math> and <math>\lambda</math>, determine the grating constant <math>a</math> of Sample 3 in meters to three significant figures for each diffraction order from the 1st order (<math>m = 1</math>) up to the 6th order (<math>m = 6</math>) [<i>Hints: These orders correspond to the six spots above the zero-order spot</i>]. Enter your results in the corresponding table in the answer sheet.</p>	1.8
D4	<p>Calculate the mean and the standard error of the mean for the grating constant <math>a</math> in meters to three significant figures. Enter your results in the corresponding table in the answer sheet.</p>	0.8

**Solutions of D2-D4** involve solving a quadratic equation of “a” and getting the its mean value and standard error of the mean → straightforward!



## Part E: Determination of the unknown angle $\phi^*$ for Sample 4

**Sample 4: a steel piece with unknown angle  $\phi^*$**



Observed reflected diffraction on the observation board

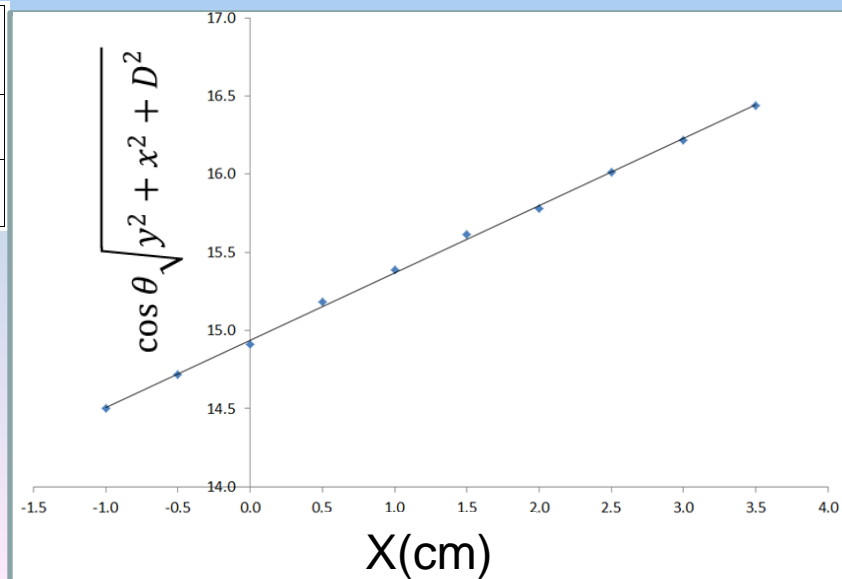
## Tasks E1-E2

Tasks		Marks
E1	Along the continuous diffracted curve of Sample 4 projected on the graph paper, measure the $y$ -coordinates in cm for ten points starting from $x = -1.0$ cm to 3.5 cm with a step of 0.5 cm. Enter your results in the corresponding table in the answer sheet.	0.3
E2	<p>Based on Eq. (1) given in Task D, construct a linear equation in the form of</p> $M(y, x, D, \theta) = I(D) + S(\phi^*)x \quad (4)$ <p>Determine the functional forms for <math>M(y, x, D, \theta)</math>, <math>I(D)</math> and <math>S(\phi^*)</math>. Plot <math>M</math> against <math>x</math>, using the data recorded in E1. Determine the unknown angle <math>\phi^*</math> in degrees from this graph. Write down all the functional forms and the value of <math>\phi^*</math> in the corresponding table in the answer sheet.</p>	1.3

$x$ co-ordinate (cm)	$y$ co-ordinate (cm)
-1.0	3.5
-0.5	4.5
0.0	5.2
0.5	6.0
1.0	6.5
1.5	7.0
2.0	7.3
2.5	7.7
3.0	8.0
3.5	8.3

$M(y, x, D, \theta)$	$\cos \theta \sqrt{y^2 + x^2 + D^2}$
$I$	$D$
$S$	$\tan \phi^*$

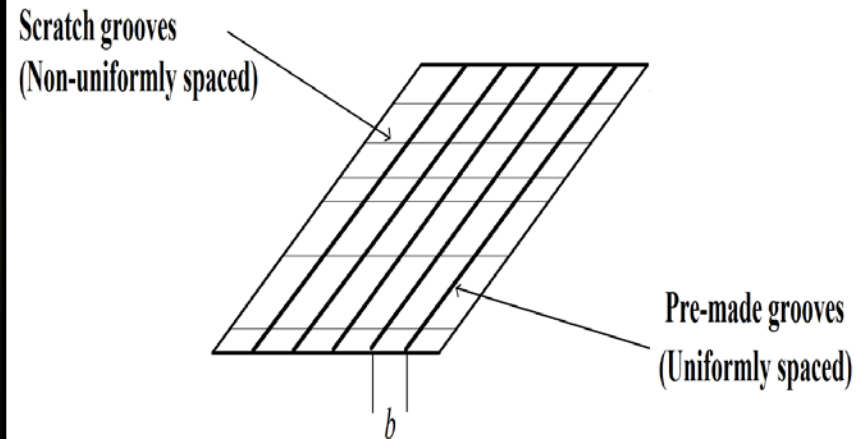
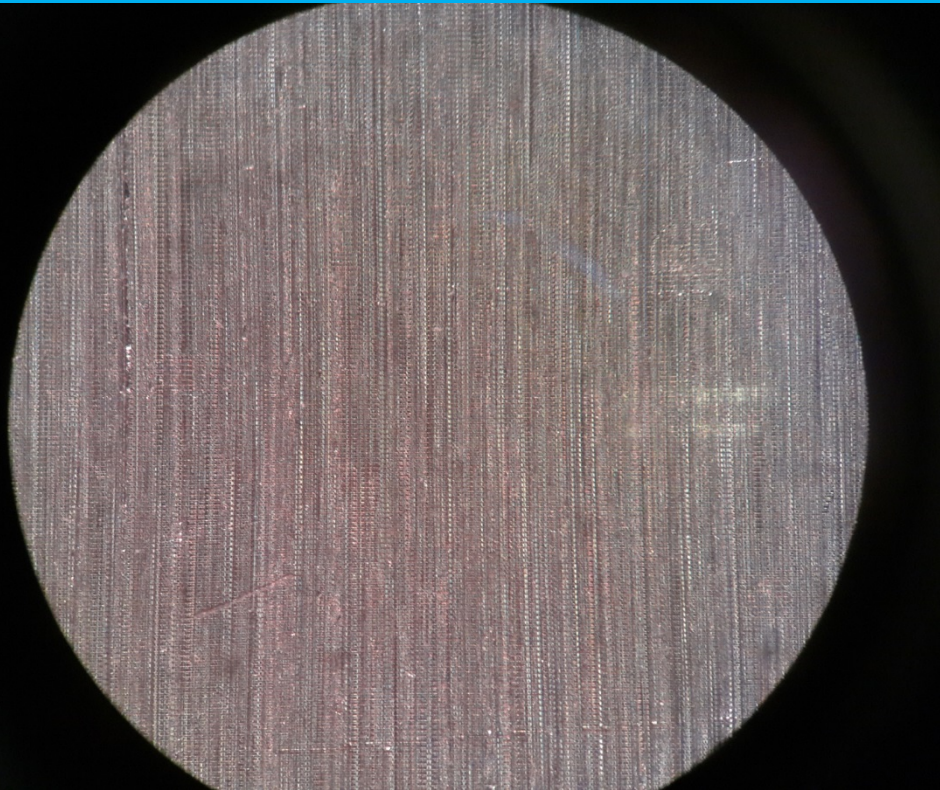
$$\phi^* = 23.2^\circ$$





## Part F: Diffraction patterns from Sample 5

**Sample 5: a steel plate with non-uniformly spaced scratch grooves + uniformly spaced grooves perpendicular to the scratch grooves (photoresist pattern)**



**Schematic drawing of Sample 5**

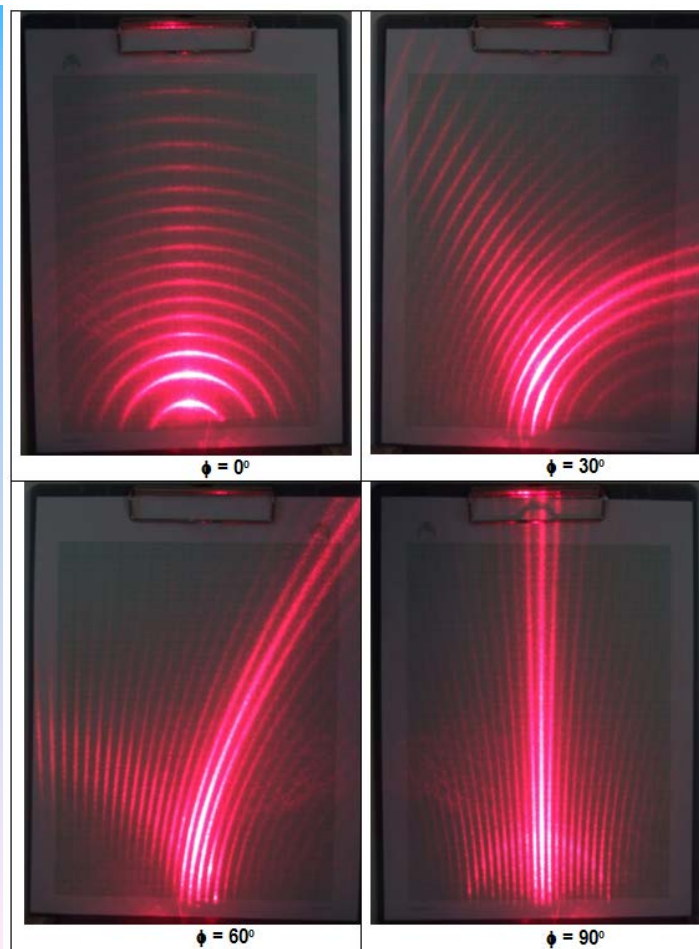
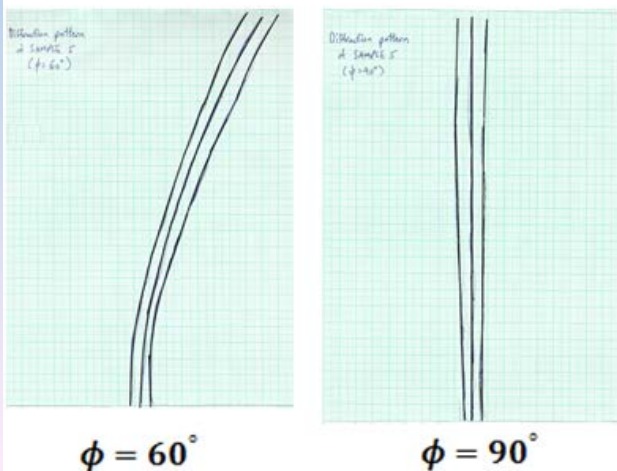
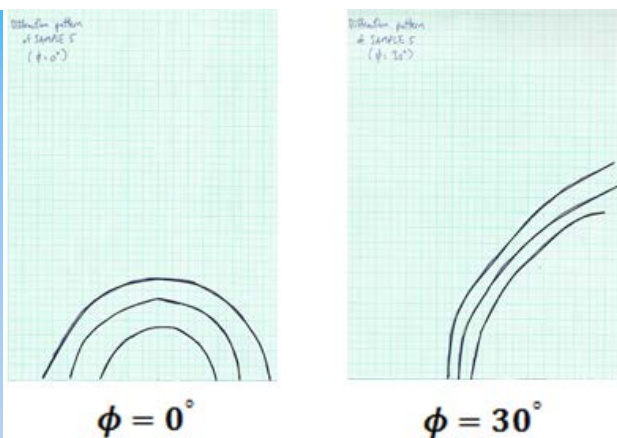
**Sample 5 viewed under microscope**

**Sample 5 is the optical analogy of the nano-grating ZnSe surface !**



# Tasks F1

Tasks		Marks
F1	Record the diffraction patterns you observed for $\phi = 0^\circ$ , $30^\circ$ , $60^\circ$ and $90^\circ$ on separate graph papers for each value of $\phi$ . At the top of each graph paper, put down '#5' and the corresponding $\phi$ value. It is expected that you could observe more than 10 diffraction orders. However, you are required to record only three relatively brighter orders on each graph paper.	0.8

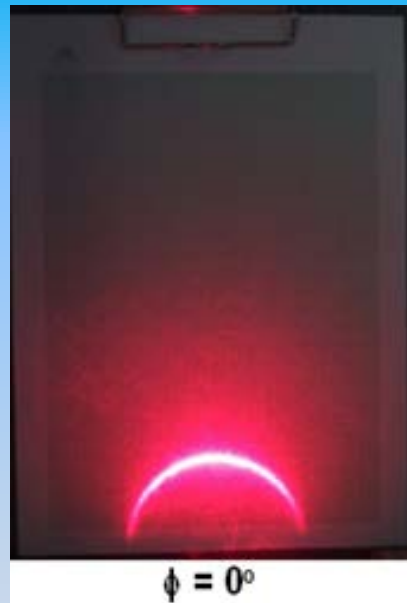




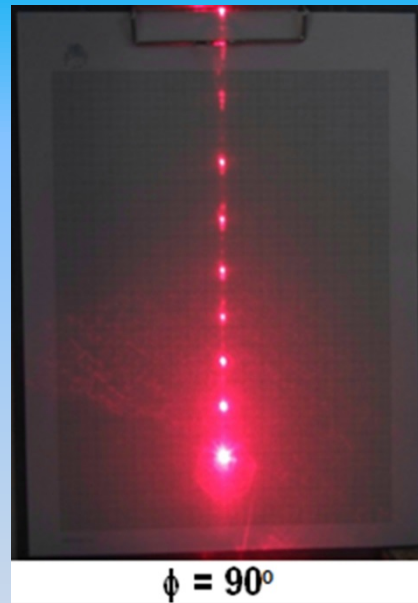


## Task F2

Tasks		Marks
F2	<p>With this understanding, estimate the spacing <math>b</math> in meters of the uniformly spaced pre-made grooves of Sample 5 using the recorded diffraction pattern for <math>\phi = 0^\circ</math> from Task (F1). Enter the value of <math>b</math> in the answer sheet.</p> <p><i>[Note that in estimating the value of <math>b</math>, you are only required to take the measured data of the first diffraction order and the estimated <math>b</math> should be rounded up to three significant figures.]</i></p>	1.2



×



1<sup>st</sup> order  
diffraction  
arc



$\phi = 0^\circ$  for non-uniformly spaced grooves;

$\phi = 90^\circ$  for uniformly spaced grooves;

The  $y_1$  value of the peak of the first order diffraction arc should satisfy the same relation as the solution of Task D:

$$y_1 = D \sqrt{\frac{b^2}{(b \cos \theta - \lambda)^2} - 1}$$

where  $b$  is the spacing of the pre-made grooves. From the recorded diffraction at  $\phi = 0^\circ$  for Sample 5, the value of  $y_1$  is measured to be 0.0695 m. Similar to what has been done in Task (D1), one can rearrange the equation for  $y_1$  to form a quadratic equation as

$$Ab^2 + Bb + C = 0$$

Where

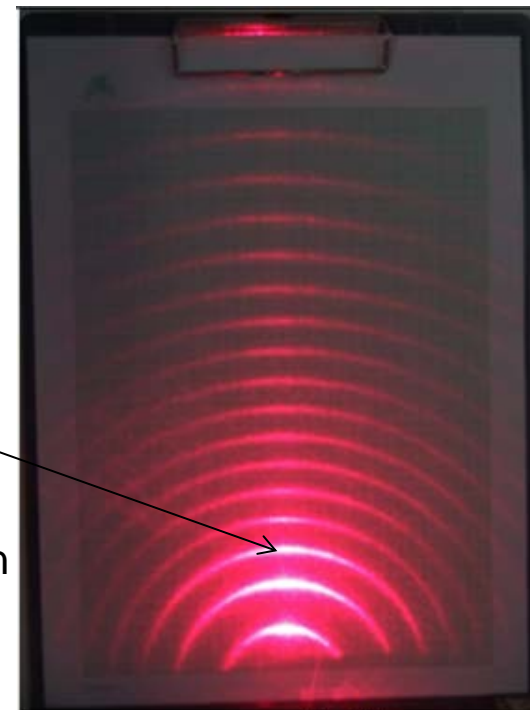
<b>A</b>	$y_1^2 \cos^2 \theta + D^2 \cos^2 \theta - D^2 = 1.481 \times 10^{-3}$
<b>B</b>	$-2\lambda \cos \theta (y_1^2 + D^2) = -3.320 \times 10^{-8}$
<b>C</b>	$\lambda^2 (y_1^2 + D^2) = 1.149 \times 10^{-14}$

Again, we take the only valid solution of  $b$  (See the solution of Task (D3) for detailed explanations) as

$$b = \frac{-B + \sqrt{B^2 - 4AC}}{2A}$$

$$\therefore b \approx 2.21 \times 10^{-5} \text{ m}$$

1<sup>st</sup> order  
diffraction  
arc

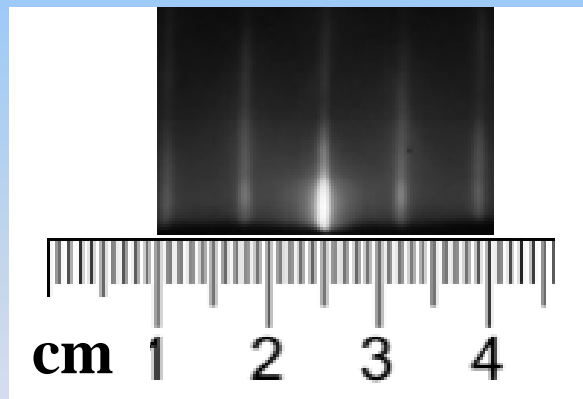
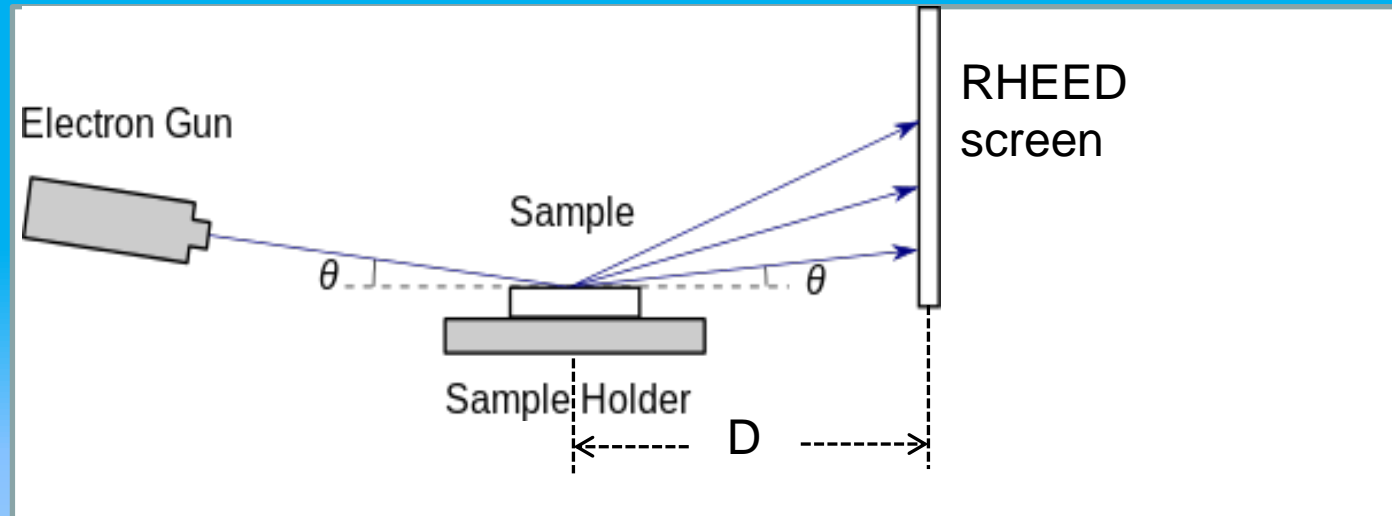


$\phi = 0^\circ$



## Part G: Determination of the lattice-plane spacing for ZnSe

Schematic diagram of the RHEED diffraction geometry



**Figure 16:** The reflection high energy electron diffraction (RHEED) pattern from a nano-structured surface of ZnSe, when the electron-beam is perpendicular to the nano-grooves with non-uniform spacing.

Given 
$$\lambda = \frac{12.247 \times 10^{-10}}{\sqrt{V(1 + 10^{-6}V)}} \text{ [m]}$$

$V = 13,000 \text{ volts}, \theta \approx 0^\circ \text{ and } D = 26 \text{ cm}$

where  $\lambda$  is the wavelength of the incident electrons and  $V$  is the accelerating voltage.

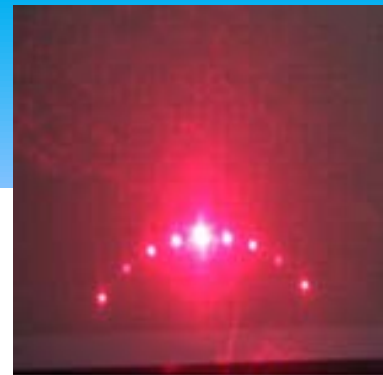
# Tasks G1

Tasks		Marks
G1	For the ZnSe sample, based on Figure 16 and the experimental conditions given above, determine the lattice-plane spacing $a^*$ of the periodic atomic lattice planes that are perpendicular to the nano-grooves with non-uniform spacing, in meters. Enter your result in the corresponding table in the answer sheet.	1.3

**Solution:**

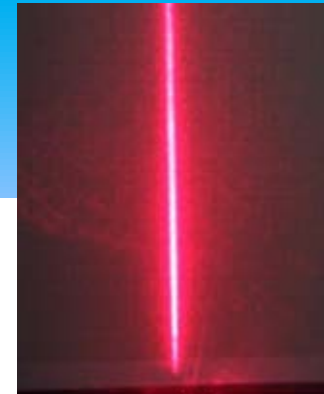
Recalling Eq. (2) given in Task (D),

$$x = \frac{Dm\lambda \cos \phi}{a^* \cos \theta - m\lambda \sin \phi}$$



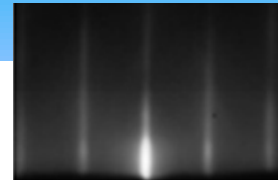
$\phi = 0^\circ$

×



$\phi = 90^\circ$

→



For the periodic atomic lattice planes, one has  $\theta \approx 0^\circ$  and  $\phi = 0^\circ$  and so Eq. (2) becomes

$$x = \frac{Dm\lambda}{a^*}$$

where  $a^*$  is the lattice plane spacing of the periodic atomic lattice planes that are perpendicular to nano-grooves with non-uniform spacing. Thus the average spacing of the RHEED streaks can be written as

$$\Delta x = \frac{D\lambda}{a^*}$$

## Task G1 continue

$\Delta x$  can be measured from the given RHEED pattern to be 0.7 cm. Given that  $D = 0.26$  m and  $\lambda$  can be calculated using the given formula to be  $0.1067 \times 10^{-10}$  m. Thus, the required lattice plane spacing of ZnSe can be calculated as

$$a^* = \frac{0.26 \text{ m} \times (0.1067 \times 10^{-10} \text{ m})}{0.007 \text{ m}}$$

$$\therefore a^* = 3.96 \times 10^{-10} \text{ m}$$

Remark: For ZnSe, the actual plane spacing for the corresponding lattice plane is  $a^* = 4 \times 10^{-10} \text{ m}$ .