FABRICATION AND CHARACTERISATION OF ZINC SULFIDE OPTICAL WAVEGUIDES USING THERMOELECTRICALLY COOLED SUBSTRATE HOLDER

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SCHOOL OF SCIENCE AND TECHNOLOGY
UNIVERSITI MALAYSIA SABAH
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FABRICATION AND CHARACTERISATION OF ZINC SULFIDE OPTICAL WAVEGUIDES USING THERMOELECTRICALLY COOLED SUBSTRATE HOLDER

ABSTRACT

This study describes the fabrication and the characterisation of zinc sulfide optical waveguides produced by thermal evaporation technique at low substrate temperatures. The substrate temperature of particular interest was –50°C because of lowest propagation loss as reported in literature. A novel thermoelectrically cooled substrate holder was designed and constructed for substrate cooling. In the first instance, a series of ambient deposited ZnS waveguide with the nominal thicknesses of 0.15 µm, 0.50 µm and 0.80 µm were fabricated on oxidised silicon wafer. The propagation modes of these samples are measured with a prism coupler. The propagation modes were analysed to verify the thicknesses and to determine the refractive index. The thicknesses from the modal analyses were found matching well with both nominal thicknesses and measured thicknesses. The refractive index of ambient deposited ZnS waveguide were increased with the increase of sample thicknesses. Based on these introductory experiments, the fabrication of 0.50 µm thick ZnS waveguides were performed at ambient temperature (25°C) and cold substrate temperature (–50°C). The ambient deposited ZnS waveguide was microcrystalline with the preferred plane of (111) whereas the cold deposited ZnS waveguide was amorphous. The grain size of the ambient deposited ZnS waveguide was about three times bigger than the cold deposited ZnS waveguide. The AFM images of the waveguides revealed that the growth mechanism of ZnS thin films was through island growth. The surface images of ZnS waveguides were obtained with an atomic force microscopy. The surface of cold deposited ZnS waveguide was rougher than ambient deposited ZnS waveguide. The propagation losses of ZnS waveguides were determined by a scattering detection method. Propagation losses of cold deposited ZnS waveguide were 20.41, 11.35, 3.51 and 2.30 dB/cm measured the wavelengths of 633, 986, 1305 and 1540 nm, respectively. Propagation losses of ambient deposited ZnS waveguide were 131.50, 47.99, 4.43 and 2.74 dB/cm measured the wavelengths of 633, 986, 1305 and 1540 nm, respectively. The wavelength dependence of propagation losses analyses were done to establish the loss mechanisms in ZnS waveguides. The results showed that the propagation loss of ambient deposited ZnS waveguide had a $\lambda^{-4.6}$ dependence and the propagation loss of cold deposited ZnS waveguide had a $\lambda^{-2.5}$ dependence. The propagation loss of the ambient deposited ZnS waveguide was dominated by bulk scattering (Rayleigh scattering) whereas the propagation loss of the cold deposited ZnS waveguide was dominated by surface scattering.

ABSTRAK

Kajian ini memperihalkan fabrikasi dan pencirian bagi pandugelombang optik zink sulfida yang dihasilkan melalui teknik penyejatan terma pada suhu substrak yang rendah. Suhu substrak $-50^{0}C$ menjadi perhatian kerana kehilangan perambatan adalah terendah berpandukan laporan daripada literatur. Satu pemegang substrak yang disejukkan oleh peranti termoelektrik yang baru telah direkabentuk dan dibina untuk penyejukkan substrak. Pada permulaannya, satu siri sampel percubaan pandugelombang ZnS dimendapkan pada suhu sekeliling dengan ketebalan anggaran 0.15 µm, 0.50 µm dan 0.80 µm telah difabrikasikan di atas substrak wafer silikon oksida. Mod perambatan sampel-sampel tersebut diukur dengan menggunakan satu penganding prisma. Mod perambatan dianalisa untuk mengesahkan ketebalan dan mendapatkan nilai indeks biasan. Ketebalan daripada analisa mod tersebut didapati berpadanan dengan ketebalan anggaran dan juga ketebalan yang diukur. Indeks biasan pandugelombang ZnS yang dimendapkan pada suhu sekeliling didapati meningkat dengan peningkatan ketebalan sampel. Berdasarkan eksperimen awal ini, fabrikasi pandugelombang ZnS berketebalan 0.50 µm dibuat pada suhu sekeliling ($25^{0}C$) dan suhu substrak sejuk ($-50^{0}C$). Pandugelombang ZnS suhu sekeliling adalah mikro-kristal dengan satah terpilih (111) manakala pandugelombang ZnS suhu sejuk adalah amorfus. Saiz butiran pandugelombang ZnS suhu sekeliling adalah lebihkurang tiga kali lebih besar daripada butiran pandugelombang ZnS suhu sejuk. Imej-imej AFM pandugelombang menunjukkan mekanisma pertumbuhan filem nipis ZnS adalah melalui pertumbuhan pulau. Imej permukaan pandugelombang ZnS diambil dengan mikroskopi daya atomik. Permukaan pandugelombang ZnS yang dimendapkan pada suhu sejuk didapati lebih kasar berbanding pandugelombang ZnS yang dimendapkan pada suhu sekeliling. Kehilangan perambatan bagi pandugelombang ZnS diperolehi dengan kaedah pengesanan penyerakkan. Kehilangan bagi pandugelombang ZnS suhu sekeliling adalah 131.50, 47.99, 4.43 dan 2.74 dB/cm yang diukur dengan jarakgelombang masing-masing 633, 986, 1305 dan 1540 nm. Kehilangan bagi pandugelombang ZnS suhu sejuk adalah 20.41, 11.35, 3.51 dan 2.30 dB/cm yang diukur dengan jarakgelombang masing-masing 633, 986, 1305 dan 1540 nm. Keputusan menunjukkan kebergantungan kehilangan perambatan bagi pandugelombang ZnS suhu sekeliling adalah $\lambda^{-4.6}$ manakala untuk pandugelombang ZnS suhu sejuk adalah $\lambda^{-2.5}$. Kehilangan perambatan dalam pandugelombang ZnS suhu sekeliling didominasi oleh penyerakan dalaman (Penyerakkan Rayleigh) manakala untuk kehilangan dalam pandugelombang ZnS suhu sejuk didominasi oleh penyerakkan permukaan.

TABLE OF CONTENTS

TITLE i
DECLARATION ii
CERTIFICATION iii
ACKNOWLEDGEMENTS iv
ABSTRACT v
ABSTRAK vi
TABLE OF CONTENTS vii
LIST OF TABLES ix
LIST OF FIGURES x
LIST OF ABBREVIATIONS AND SYMBOLS xi
LIST OF APPENDICES xii

CHAPTER 1 INTRODUCTION

1.1 ZnS Thin Films 1
1.2 Thermally Deposited ZnS Thin Films 3
1.3 Cold Deposited ZnS Waveguides 5
1.4 The Problem Statement and the Originality of Study 6
1.5 Objectives 8
1.6 The Layout of the Thesis 9

CHAPTER 2 REVIEW ON ZnS THIN FILM DEPOSITION TECHNIQUES

2.1 Introduction 11
2.2 Chemical Deposition Techniques 11
  2.2.1 Chemical Vapour Deposition 12
  2.2.2 Chemical Bath Deposition 13
  2.2.3 Sol-Gel Technique 14
  2.2.4 Spray Pyrolysis 15
2.3 Physical Deposition Techniques 16
  2.3.1 Sputtering 16
  2.3.2 Pulsed Laser Deposition 17
  2.3.3 Molecular Beam Epitaxy 19
2.4 Thermal Evaporation Technique 21
  2.4.1 The Theory of Thermal Evaporation 22
  2.4.2 Cold Deposition Methods 24

CHAPTER 3 THIN FILMS BASICS AND THE THEORY OF WAVEGUIDE

3.1 Introduction 27
3.2 The Basics of Thin Films 27
  3.2.1 Growth and Nucleation 28
  3.2.2 The Microstructure and the Surface Roughness of Thin Films 30
3.3 The Theory of Planar Waveguide 31
  3.3.1 Ray-Optical Waveguide Theory 31
  3.3.2 The Wave Equations and Their Solutions 37
3.4 Loss Mechanisms in Optical Waveguides 41

CHAPTER 4 DESIGN AND CONSTRUCTION OF NOVEL THERMOELECTRICALLY COOLED SUBSTRATE HOLDER

4.1 Introduction 43
4.2 Background of the Thermoelectric Devices 43
4.3 Description of the Substrate Holder Design 46
4.4 Temperature Validation Method 50
4.5 Cooling Performance of the Substrate Holder 51
4.6 Summary 53

CHAPTER 5 EXPERIMENTAL SET UP AND PROCEDURES

5.1 Introduction 54
5.2 ZnS Thin Film Deposition 54
  5.2.1 The Vacuum System 54
  5.2.2 Substrate Preparation 56
  5.2.3 Deposition Procedures 58
5.3 Characterisation Techniques 61
  5.3.1 Atomic Force Microscopy 61
  5.3.2 X-Ray Diffractometer 64
  5.3.3 Prism Coupler 66
  5.3.4 Scattering Detection Method 68
  5.3.5 Multiple-Wavelength Measurement Setup 70

CHAPTER 6 EXPERIMENTAL RESULTS AND DISCUSSION

6.1 Introduction 72
6.2 Thickness and Propagation Mode of the Trial Samples 73
6.3 Structural Characteristics of ZnS Waveguides 78
6.4 Grain Size and Surface Roughness of ZnS Waveguides 81
6.5 Propagation Loss of ZnS Waveguides 87
6.5 Wavelength Dependence of Propagation Loss 93

CHAPTER 7 CONCLUSION AND SUGGESTION

7.1 Conclusion 97
7.2 Suggestions for Future Research 100

REFERENCES **102**

APPENDICES **109**

LIST OF TABLES

		Page
Table 1.1	Thermally evaporated ZnS thin films.from selected literatures.	4
Table 4.1	Validation of substrate temperature measurements.	50
Table 5.1	ZnS deposition parameters.	60
Table 6.1	The thicknesses and refractive index of ZnS waveguide trial samples.	76
Table 6.2	Comparisons of standard value of ZnS and observed values for ambient and cold deposited ZnS waveguides.	80
Table 6.3	Comparisons of crystallite size of the ambient deposited ZnS waveguide and the grain sizes of ambient and cold deposited ZnS waveguides.	84
Table 6.4	Surface roughness of ambient and cold deposited ZnS waveguide.	86
Table 6.5	Propagation losses of ZnS waveguides.	92

LIST OF FIGURES

Page

Figure 1.1 ZnS waveguide loss as a function of substrate temperature. 6

Figure 2.1 Schematic of the basic apparatus for DC and RF sputtering. 16

Figure 2.2 Schematic of the apparatus for PLD. 18

Figure 2.3 Schematic of MBE deposition chamber. 21

Figure 2.4 Flux passing small area source to elemental substrate. 24

Figure 2.5 Schematic drawing of the nitrogen cooled substrate holder. 26

Figure 3.1 Schematic of the stages of thin film growth. 27

Figure 3.2 Schematic of three basic modes thin film growth. 28

Figure 3.3 Guided light (ray) inside a waveguide structure. 32

Figure 3.4 The relationship between β and the other propagation vectors. 33

Figure 3.5 Geometric construction for calculating the phase shift. 35

Figure 4.1 Thermoelectric effects in metal-semiconductor junctions. 44

Figure 4.2 Cross section of a typical thermoelectric device. 45

Figure 4.3 Schematic drawing of thermoelectrically cooled substrate holder. 47

Figure 4.4 Photograph of the thermoelectrically cooled substrate holder. 48

Figure 4.5 Temperature dependence of an oxidised silicon substrate on the thermoelectrically cooled substrate holder. 52

Figure 5.1 Schematic diagram of the thermal evaporation system with the thermoelectrically cooled substrate holder placed under the top cover. 55

Figure 5.2	The detection system of a typical AFM.	62
Figure 5.3	The schematic diagram of AFM.	63
Figure 5.4	The schematic of X-ray diffractometer apparatus.	65
Figure 5.5	The experimental arrangement for prism coupling excitation.	67
Figure 5.6	Scattering detection method for propagation loss measurement.	69
Figure 5.7	The experimental set up for the multi-wavelength measurement.	71
Figure 6.1	Measured light intensity (TE polarised) versus incident angle for the 0.15 μm ZnS waveguide trial sample.	74
Figure 6.2	Measured light intensity (TE polarised) versus incident angle for the 0.50 μm ZnS waveguide trial sample.	75
Figure 6.3	Measured light intensity (TE polarised) versus incident angle for the 0.80 μm ZnS waveguide trial sample.	75
Figure 6.4	XRD diffractogram of the ambient deposited ZnS waveguide.	79
Figure 6.5	XRD diffractogram of the cold deposited ZnS waveguide.	79
Figure 6.6	2-D AFM micrography and surface cross section of ambient deposited ZnS waveguide.	81
Figure 6.7	2-D AFM micrography and surface cross section of cold deposited ZnS waveguide.	82
Figure 6.8	3-D AFM micrography of ambient deposited ZnS waveguide with scan area of 3.5 μm X 3.5 μm.	85
Figure 6.9	3-D AFM micrography of cold deposited ZnS waveguide with scan area of 3.5 μm X 3.5 μm.	85
Figure 6.10	Propagation loss of ZnS waveguides measured with λ_1 = 633 nm (a) deposited at ambient temperature and (b) deposited at cold temperature.	88

Figure 6.11 Propagation loss of ZnS waveguides measured with λ_2 = 986 nm (a) deposited at ambient temperature and (b) deposited at cold temperature. 89

Figure 6.12 Propagation loss of ZnS waveguides measured with λ_3 = 1305 nm (a) deposited at ambient temperature and (b) deposited at cold temperature. 90

Figure 6.13 Propagation loss of ZnS waveguides measured with λ_4 = 1540 nm (a) deposited at ambient temperature and (b) deposited at cold temperature. 91

Figure 6.14 Propagation loss versus wavelength for ambient and cold deposited ZnS waveguides. 94

LIST OF ABBREVIATIONS AND SYMBOLS

α	propagation loss, evaporation coefficient
β	propagation constant, FWHM of diffraction peak
λ	wavelength of the light
μm	micrometer
AFM	atomic force microscopy
CBD	chemical bath deposition
CCD	charge couple device
CVD	chemical vapour deposition
d	grain size
dB/cm	decibel per centimetre
D	crystallite size
DC	direct current
eV	electron volt
FWHM	full width at half maximum
HeNe laser	helium neon laser
l/min	liter per minute
l/s	liter per second
mTorr	miliTorr
M	number of propagation mode
MBE	molecular beam epitaxy
nm	nanometer
nm/s	nanometer per second
n_{Ox}	refractive index of silicon dioxide
n_{Si}	refractive index of silicon
n_{ZnS}	refractive index of ZnS
^{o}C	degree Celsius
PLD	pulsed laser deposition
RF	radio frequency
rms	root mean square
TE	transverse electric
TEC	thermoelectric coolers
TM	transverse magnetic
T_S	substrate temperature
V	volt

LIST OF APPENDICES

		Page
APPENDIX A	UNCERTAINTIES OF THE EQUIPMENTS	109
APPENDIX B	CALCULATION OF NUMBER OF THE PROPAGATION MODES	110
APPENDIX C	ESTIMATION OF DEPOSITION TIME	111
APPENDIX D	LIST OF PUBLICATIONS	112
APPENDIX E	VERIFICATION OF SILICON DIOXIDE THICKNESS	113

CHAPTER 1

INTRODUCTION

This thesis will discuss a study on the fabrication processes and the properties of zinc sulfide (ZnS) planar optical waveguides. A commercial vacuum evaporator was used to deposit ZnS thin films at relatively low substrate temperatures. The substrate temperatures during the deposition processes were controlled at ambient temperature (25^{0}C) for ambient deposition and cold temperature (-50^{0}C) for cold deposition. The substrate cooling in cold deposition was achieved using a self-invented thermoelectrically cooled substrate holder. The substrate holder assembly that was designed for this study was made up of one of only two substrate cooling techniques used for the thermal deposition below freezing point. This represents the first ZnS waveguide fabricated using thermoelectrically cooled substrate holder in vacuum chamber.

1.1 ZnS Thin Films

ZnS is a white to yellow coloured powder or crystal. It naturally exists in the more stable cubic form, known as the mineral sphalerite (β-ZnS). The less stable form i.e. the hexagonal form of ZnS, however, is a synthetic material and is also known as the mineral wurtzite (α-ZnS). Both sphalerite and wurtzite form of ZnS are II-VI compound semiconductors with a wide direct energy band gap. The optical bandgap of α-ZnS and β-ZnS at 300 K is 3.8 eV and 3.6 eV (Nadeem *et al.*, 2005).

ZnS has a high index of refraction of 2.35 (at wavelength of 550 nm) and is transparent over a very broad range (0.4 μm up to 13 μm). The electronic absorption edge at 0.33 μm corresponds to an energy band gap of 3.7 eV. These optical properties make ZnS thin films useful in filters and reflectors. ZnS also have a high potential to used as waveguide that can be integrated into optical components.

ZnS thin films are widely used as thin film dielectric coatings in optical and microelectronics industries. In the area of optics, ZnS thin films have been used as reflectors and dielectric filters because of their high refractive index and high transmittance in the visible range. This extraordinary properties make ZnS an important material for light propagation medium or waveguiding material. With a high index value, it is possible to deposit a relatively thin waveguide of ZnS that can support multiple modes of propagation.

ZnS thin film is a promising material for use in various application devices. Monochrome thin film electroluminescent (TFEL) panels based on ZnS doped with manganese (Mn) phospors have been commercially available for several years (Wilson and Hawkes, 1998). A considerable amount of work has been done out to produce different colours of electroluminescent display using ZnS thin films as host material. Mn is also used as an active species to produce a characteristically yellow-orange emission at 590 nm (Tang and Cameroon, 1996). Wider range of colours were obtained by using rare earth elements. Red, blue and green emissions have been obtained using samarium (Sm), thulium (Tm) and terbium (Tb) respectively.

In opto-electronics, ZnS thin films were used as light emiting diode (LED) for the blue to ultraviolet spectral region due to its wide bandgap at room temperature (Singh, 1996). Cuprum (Cu) doped ZnS thin films was used as the emitting layer of LED and the blue light of electroluminescence was observed at room temperature with turn-on voltage below 4 volt (Huang *et al*, 1997).

ZnS was also used as laser host materials for near- and mid-infrared lasers (Carrig, 2002). Studies on chromium (Cr) doped ZnS laser which emits in the region of two micrometer to three micrometer was first reported by DeLoach and colleages at Lawrence Livermore National Laboratory (DeLoach *et al.*, 1996). Since then, this laser material have been continously developing (Sorokina *et al.*, 2002).

1.2 Thermally Deposited ZnS Thin Films

Thin films grown by any particular technique may have different properties due to the involvement of various deposition parameters. It is therefore, essential to make a detailed investigation on the relationship between the properties of these films and the methods of deposition. ZnS thin films have been deposited using many techniques. This section, however, is limited to discussing the thermally deposited ZnS thin films only. The sructures and properties of ZnS thin films deposited using other techniques are presented in Chapter 2 together with the details of each techniques.

The microstructure of deposited thin films can usually be described by either an amorphous or polycrystalline structures. A polycrystalline film has a measurable grain size but the amorphous film is grainless. The crystallite grain size is dependent on adatom surface mobility that increases with substrate temperature. At high temperature, adatoms become very active and have enough energy to combine to each other to form larger grains. Therefore, a decrease in substrate temperature during deposition should result in smaller grains. ZnS thin films deposited onto amorphous substrate at or above ambient temperatures normally have polycrytalline structures.

The temperature of the substrate plays a vital role in determining the structure of an amorphous or polycrystalline for the thermally deposited thin films. In general, covalently bonded materials such as semiconductors produced either amorphous structures at low substrate temperatures or polycrystalline structures at higher temperatures or epitaxial single crystal structures under the same conditions of high temperature deposition (Thornton, 1988).

ZnS thin film prepared by thermal evaporation will possibly exhibit waveguide characteristics that is indicated by light (power) propagation losses. The propagation loss is related to the structure of the waveguide, and in turn is affected by substrate temperature. Table 1.1 is a summary of the selected reports on thermally evaporated ZnS thin films from literatures. Propagation losses stated in the table were measured with He-Ne laser at the wavelength of 633 nm.

Table 1.1: Thermally deposited ZnS thin films from selected literatures. (*Propagation loss listed here were measured with laser at 633 nm*)

Substrate	**Thermal source**	**Substrate temperature (^{o}C)**	**Film structure and *propagation loss***	**Reference**
Oxidised silicon wafer	Alumina crucible	Cold (–50) and ambient (25)	Amorphous and microcrystalline, *131.50 dB/cm and 20.41 dB/cm. (Losses at 986, 1305 & 1540 nm are presented)*	**This study**
Quartz	Not mentioned	Room temperature(*)	Polycrystalline, loss not measured.	Wu *et al.* (2008)
Corning 7059 glass	Mo boat	Hot (300)	Polycrystalline, loss not measured	Pratap *et al.* (2008)
Corning 7059 glass	Boat	Room temperature (27)	Polycrystalline, loss not measured.	Reddy *et al.* (2007)
Corning 7059 glass	Boat	Hot (200 to 350)	Polycrystalline, loss not measured.	Subbaiah *et al.* (2006)
Silica glass	Mo boat	Ambient temperature(*)	Polycrystalline, loss not measured.	Durrani *et al.* (2000)
Quartz, oxidised silicon wafer	Mo boat	Hot (150)	Polycrystalline, loss not measured.	Huang (1999)
Glass, oxidised silicon wafer	Mo boat	Ambient temperature(*)	Polycrystalline, loss not measured.	Zhao-Hong *et al.* (1998)
Oxidised silicon wafer	Alumina crucible	Ambient temperature(*)	Polycrystalline, *6 to 40 dB/cm.*	Himel and Kimble (1993)
Oxidised silicon wafer	Ta boat	Ambient temperature(*)	Polycrystalline, loss not measured.	Wong *et al.* (1991)
Silica glass, oxidised silicon wafer	Alumina crucible	Cold to Moderate (–120 to 50)	Amorphous to microcrystalline, *1 to 100 dB/cm.*	Ruffner *et al.* (1989)
Quartz, Oxidised silicon wafer	Alumina crucible	Ambient temperature(*)	Polycrystalline, *8.0 to 16.5 dB/cm.*	Himel *et al.* (1987)
Oxidised silicon wafer	Alumina crucible	Ambient temperature(*)	Polycrystalline, *5.2 to 17.3 dB/cm.*	Himel and Gibson (1986)
Glass	Ta boat	Moderate to hot (35 to 300)	Polycrystalline, *60 to 90 dB/cm.*	Al-Douri (1986)

* actual temperature were not mentioned.

The possibility of producing ZnS waveguides with various losses by varying the substrate temperature was first reported by Al-Douri (1986). He found that the losses of the films incerased from 60 dB/cm to 90 dB/cm when the substrate temperature increased from 35^{0}C to 300^{0}C. He suggested that the propagation loss can be reduced by depositing ZnS through slow deposition rates (1 nm/s and less).

Propagation losses in ZnS waveguides deposited at lower substrate temperatures (from -120^{0}C to 30^{0}C) were studied by Ruffner *et al.* (1989). In general, when the substrate surface is kept cold, the atoms being deposited will not have enough energy to diffuse along the surface to be placed in an ordered crystal. The ZnS thin films will be in the form of amorphous when the substrate temperature is cooled down to below the freezing point.

1.3 Cold Deposited ZnS Waveguides

The major causes of propagation losses in deposited waveguides are scattering and absorption by the materials. The high loss in polycrystalline ZnS waveguides are due to the combination of surface scattering and bulk scattering at the crystalline grain boundaries (Himel and Kimble, 1993). Therefore, a small grain size is necessary for a reduction of surface roughness and allows a reduction of the scattering (Wong *et al.*, 1991). The smallest grain size of ZnS can be found in its amorphous thin films and can be prepared by depositing ZnS on the cold substrate.

The results of the study by Ruffner *et al.* (1989) as in Figure 1.1 shows the propagation losses of ZnS waveguides as a function of substrate temperature. The propagation losses were minimum when the deposition were executed at the substrate temperature of -50^{0}C. For higher deposition temperatures, the enhanced crystalinity of the film may have induced scattering centers and resulted in higher losses. Below -50^{0}C, the propagation losses were increased as a result of pinhole formation and also eventually tensile stress failure occurs with decreasing deposition temperatures (Ruffner *et al.*, 1989).

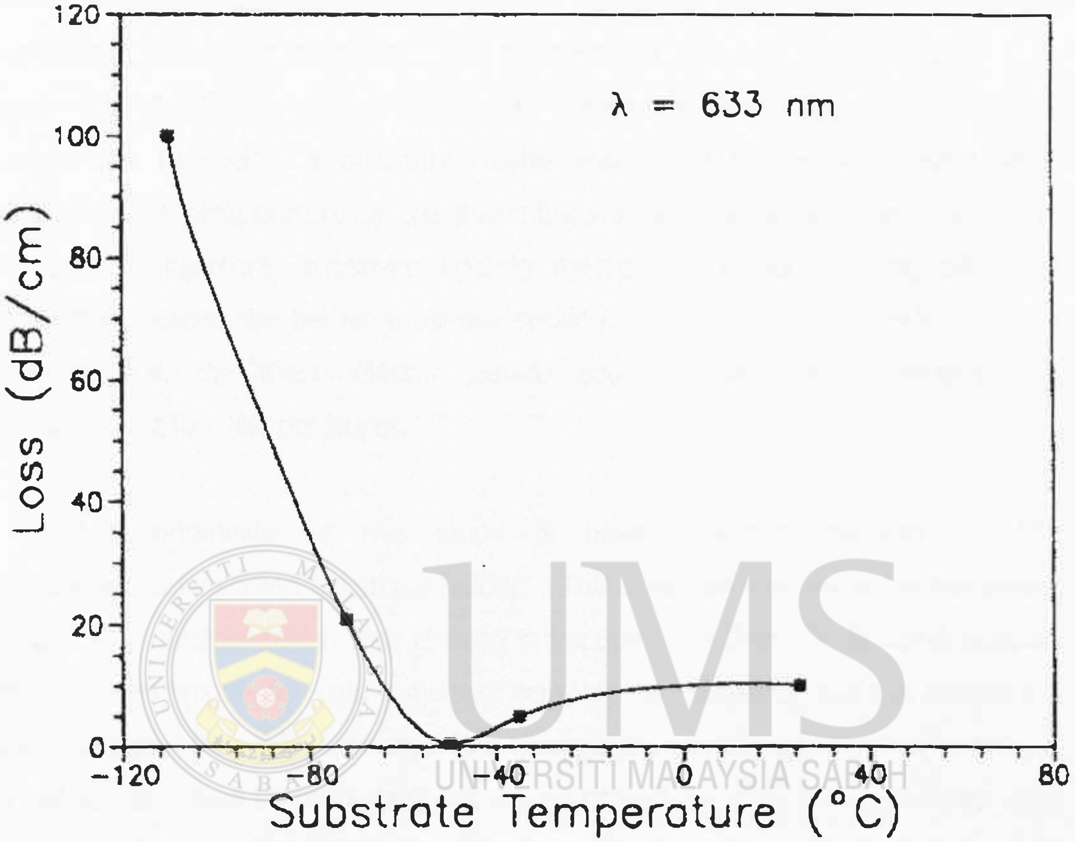

Figure 1.1: ZnS waveguide loss as a function of substrate temperature (Ruffner *et al.*, 1989).

1.4 Problem Statement and The Originality Of Study

The fabrication of ZnS waveguides using a thermal evaporator is of interest because of simple and cheap instrumentations. Literature survey indicates that the properties of thermally evaporated ZnS waveguides are acceptable and have a good prospect of utilisation but more studies need to be done to understand their properties.

Low temperature depositions of ZnS waveguides are particular aspect to be explored. Therefore, the thermal evaporation system become technically complicated because of the combination of high vacuum technology, high temperature thermal source with substrate cooling. There was no easy way of cooling the substrate down to –50°C in vacuum. Traditionally, the cooling mechanism was liquid nitrogen. The temperature of liquid nitrogen is 77 K or about –200°C and this too cold for ZnS waveguide fabrication. To bring the temperature to –50°C, a minature heater was fitted to the substrate holder. Controlling the temperature of the assembly was difficult since liquid nitrogen is not stable. Therefore, substrate cooling mechanism without relying on liquid nitrogen is needed for better substrate cooling. This project was undertaken to establish that the thermoelectric device could be utilised to deposit ZnS waveguides at low temperatures.

The originality of this study is based on the invention of the thermoelectrically cooled substrate holder. This presents the first substrate cooler using thermoelectric device that is used in vacuum chamber. It demonstrates an alternative technique for cold substrate temperture deposition without relying on liquid nitrogen. The cold deposited ZnS waveguides produced using this technique should exhibit similar characteristics as compared to the cold deposited ZnS waveguide using liquid nitrogen cooled subtrate holder.

The waveguide properties of ZnS thin films in infrared region are relatively unstudied areas. In fact, to the best of my knowledge, there is no published data on the propagation loss of ZnS waveguides at the wavelength longer than 633 nm. This study explores and provides the characteristics of ZnS waveguides at the wavelengths of 986 nm, 1305 nm, 1540 nm, giving information on the characteristics of ZnS waveguides at wavelengths significantly longer than the wavelengths that have been previously experimented upon. As a result, the wavelength dependence of the propagation loss of the ambient and cold deposited ZnS waveguides will therefore be demonstratable for each of the experimented wavelengths.

1.5 Objectives

The goal of this study is to experimentally investigate the effects of substrate cooling on the light propagation in thermally deposited ZnS waveguides which consist of substrate cooler creation and ZnS thin films deposition and characterisation. The objective of the study are divided into six equally important steps.

The first step is to design and to construct a substrate holder with a cooling element that are put together into the body of a substrate holder assembly. The cooling mechanism is achieved with a thermoelectric device which is to be supported by a liquid heat exchanger. The newly invented thermoelectrically substrate cooler built for this study is expected to be more reliable because its operational without liquid nitrogen.

The second step is to deposit ZnS thin films at cold substrate temperature (-50^{o}C) and at ambient substrate temperature (25^{o}C) using the self-built thermoelectrically cooled substrate holder. Other parameters are kept constant for every cyle of deposition i.e the same parameter values are used. The waveguide structure is obtained when ZnS thin films is deposited on oxidised silicon substrate by clamping it onto cold side of the holder assembley.

The third step is to determine the propagation modes of the trial samples with three different thickness deposited only at ambient temperature. A prism coupler is used to measure the propagation modes. The modes of propagation are then analysed in order to verify the thicknesses and to calculate the refractive indices. The thicknesses are also to be verified by direct measurement using a surface profiler.

The fourth step is to determine the crystallite phase occurrence and crystallite size of cold and ambient deposited ZnS thin films. This structural characterisation is to be studied using an X-ray diffractometer.