Jurnal Teknologi, 55 (Sains & Kej.) Keluaran Khas (1), Mei 2011: 119-128

ORGANIC-INORGANIC HYBRID SOL-GEL MATERIALS FOR OPTICAL WAVEGUIDE APPLICATION

AHMAD SHARMI ABDULLAH[1,*], AMIRJAN NAWABJAN[2], NORAZAN MOHD KASSIM[3], MOHD HANIFF IBRAHIM[4] & MOHAMAD ZAHID ABDUL MALEK[5]

Abstract Ridge optical waveguides based on organic-inorganic (hybrid) sol-gel materials were designed, simulated and fabricated. The hybrid sol-gel materials were synthesized from vinyltriethoxysilane (VTES), tetraethoxysilane (TEOS) and tetrabutoxytitanate (TTBu) precursors by means of sol-gel processing technique. The optical waveguides were fabricated on quartz substrate using spin coating, direct photolithography, and wet chemical etching techniques. Multiple layers of sol were deposited so as to obtain waveguide structure with suitable thickness for mode propagation such as acquired from the simulation. Waveguiding ability of the ridge optical waveguides at 1550 nm wavelength was characterized using direct end-face fiber butt-coupling method. Physical structure of the waveguides was observed through high power microscope. Observation showed that the proposed material possesses the ability for waveguiding application at the wavelength of 1550 nm. Simulation showed that a single mode ridge optical waveguide could be realized provided that the structure thickness and width are within certain range. The range is attainable through proper control of spin coating and micropatterning parameters. Acceptable end-face quality resulted from natural cleaving process was also discovered.

Keywords: Sol-gel processing; organic-inorganic material; ridge optical waveguide

Abstrak. Pandu gelombang optik jalur berasaskan bahan-bahan sol-gel organik-bukan organic (hybrid) direka, disimulasi dan dibentuk. Bahan-bahan sol-gel hybrid tersebut disediakan daripada vinyltriethoxysilane (VTES), tetraethoxysilane (TEOS) dan tetrabutoxytitanate (TTBu) malalui kaedah pemprosesan sol-gel. Pandu gelombang optik tersebut telah dibentuk di atas lapisan quartz menggunakan kaedah penyalutan berputar, lithografi langsung, dan goresan kimia basah. Beberapa lapisan sol telah dibentuk demi mendapatkan struktur pandu gelombang dengan ketebalan yang sesuai untuk laluan mod cahaya sebagaimana diperolehi daripada simulasi. Kebolehan pandu gelombang untuk memandu cahaya pada panjang gelombang 1550 nm telah dicirikan mengunakan kaedah direct end-face fiber butt-coupling. Struktur fizikal pandu gelombang tersebut telah diperhatikan melalui mikroskop berkuasa

[1-5] Photonic Technology Center, Infocomm Research Alliance, Universiti Teknologi Malaysia, 81310, UTM Johor Bahru, Johor Darul Ta'azim, Malaysia
* Corresponding author: sharmi@fke.utm.my

tinggi. Keputusan yang diperolehi menunjukkan bahawa bahan yang dicadangkan boleh digunakan dalam aplikasi pemanduan gelombang optik pada panjang gelombang 1550 nm. Simulasi menunjukkan bahawa pandu gelombang jalur satu mod dapat direalisasikan jika ketebalan dan kelebaran pandu gelombang tersebut berada pada suatu julat nilai tertentu. Julat nilai tersebut dapat diperolehi melalui pengawalan parameter-parameter penyalutan berputar dan pembentukan mikro. Kualiti keratan rentas yang baik juga diperhatikan telah diperolehi melalui kaedah pembelahan semulajadi.

Kata kunci: Pemprosesan sol-gel; bahan organik-bukan organic; pandu gelombang jalur

1.0 INTRODUCTION

Sol-gel processing technique put forward an alternative way of new optical material synthesis. The material produced owns properties ranges from pure silica glass to silicone rubber [1], or between mineral and organic [2]. Moreover, sol-gel processing technique deals with low processing temperature and able to produce materials with high homogeneity and purity in several forms [3]. Besides, sol-gel materials are naturally cost-effective and viable in its implementation.

Hybrid sol-gel material is known to offer way out to the setback of its inorganic counterpart in the application of optical communication devices. Although both of them, the hybrid and inorganic sol-gel materials are promised to have high homogeneity and purity, the inorganic sol-gel materials however utilize higher temperature processing, indirect patterning, and having a low thermo-optic coefficient [4]. Additionally, the mechanical stress as a result of large amount of shrinkage during thermal treatment has made the fabrication of a thick inorganic sol-gel optical waveguide without crack impossible [5].

In contrast, organic group that is introduced within the hybrid materials can function as organic modifiers or organic networks formers. These lead to the fabrication of more flexible products and also decrease the problem of shrinkage significantly [6, 7]. Many studies have been done on hybrid sol-gel materials especially with the presence of precursor such as titania or any other refractive index modifiers [8-12]. Different types of organic components have been used therein and they resulted in optical waveguides with higher thickness, crack-free, smooth surface and tailored refractive index. It is found that the propagation loss in the optical waveguide was contributed by the existence of organic components especially the C-H overtones besides precipitation of titania and optical waveguide surface roughness [13, 14]. Therefore, organic components with shorter organic chain was expected to show lower propagation loss in optical waveguiding application [15].

The existence of C=C double bond in the organic components, together with photoinitiator and catalyst, causing the hybrid sol-gel materials to become photosensitive. As a result, optical waveguide micropatterning by means of direct photolithography technique is possible with the photosensitive hybrid sol-gel materials [16-18].

In this paper, firstly ridge optical waveguide structure was designed and simulated. The purpose was to roughly obtain ridge optical waveguide structure that could guide the optical wave at 1550 nm wavelength. General structure of a ridge optical waveguide consists of a waveguide of thickness d, width w, and refractive index n_g, on top of a substrate of refractive index n_s, and cover by a cladding with refractive index n_c as shown in Figure 1. In this work, quartz was used as substrate, hybrid sol-gel material as waveguide and air as cladding. Refractive indices of those substrate, waveguide and cladding materials are known from previous work [19] as 1.4442, 1.4887 and 1.0, respectively.

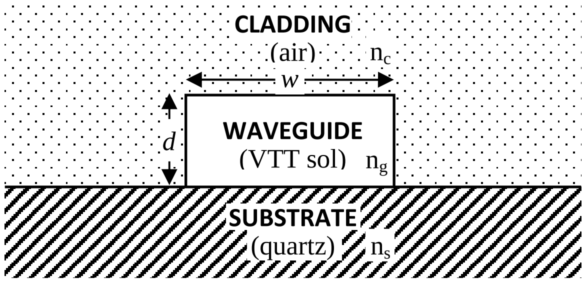

Figure 1 Cross-section of a ridge optical waveguide

Secondly, the simulated ridge optical waveguide structure was fabricated. Inorganic precursor TEOS, photosensitive organic component with shorter organic chain VTES, and refractive index modifier TTBu, were used to synthesis the hybrid VTES/TEOS/TTBu (VTT) sol. Benzoin Isobutyl Ether (BIE) and Aluminum Acetylacetone (AlAA) were used as photoinitiator and catalyst, respectively. Ridge optical waveguides were fabricated by means of multi-step spin coating, direct photolithography, and wet chemical etching techniques. Ridge optical waveguide samples were observed for their light guiding ability. The finding concluded that VTT sol-gel material can be used as optical waveguide material since it has the ability to guide light. The possibility of achieving single mode ridge optical waveguide structure at certain range of thickness and width was also simulated. Finally, the fabricated ridge optical waveguide was found to possess acceptable structure quality.

2.0 STRUCTURE DESIGN AND SIMULATION

Ridge optical waveguide structure was designed and simulated using RSoft Photonic CAD Suite BeamPROP. General structure of ridge optical waveguide such as shown in Figure 1 was implemented in the design. Refractive indices of substrate, waveguide and cladding materials are 1.4442, 1.4887 and 1.0, respectively according to the previous work [19]. The contour map of transverse index profile and its cross section are shown in Figure 2. The design was simulated for the structure cut-off condition in order to roughly obtain ridge optical waveguide structure that could work to guide the optical wave at 1550 nm wavelength.

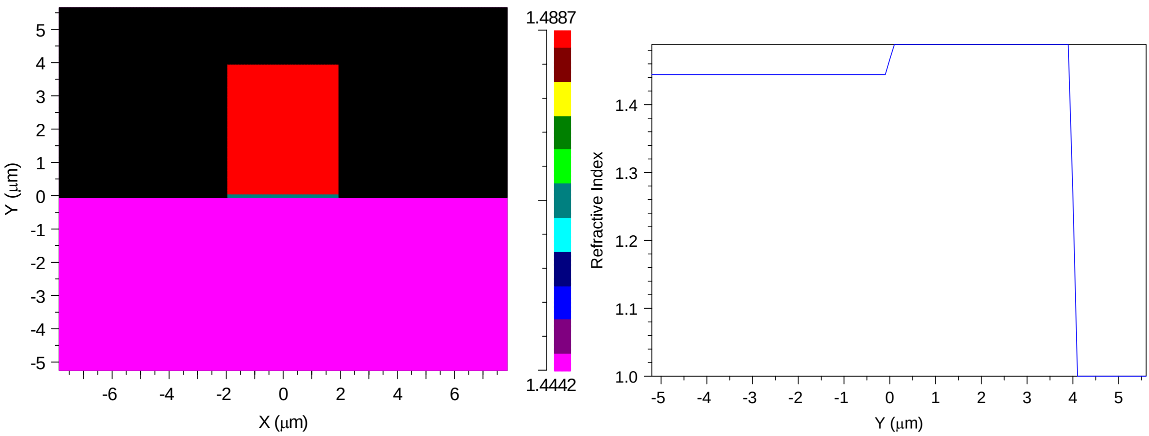

Figure 2 Contour map of transverse index profile and cross section at X = 0

3.0 HYBRID SOL-GEL MATERIALS SYNTHESIS

In this work, two compositions were prepared; (a) VTES 100% composition, and (b) TEOS:TTBu 75%:25% composition as shown in Figure 3. As for composition (a), VTES (Sigma-Aldrich, purity 99%) was hydrolyzed by deionized water (H_2O) in the presence of HCl (aq) with H_2O/VTES molar ratio of 3/2. The solution was stirred for about 4 hours before BIE and AlAA were added with VTES/BIE and VTES/AlAA molar ratios of 5/100 and 2/100, respectively. The resulted solution was then stirred for another 30 minutes.

As for composition (b), TEOS (Fluka, purity 98%) was hydrolyzed by deionized water in the presence of HCl (aq) and ethanol (EtOH) with TEOS/EtOH/H_2O molar ratio of 3/4/12. The solution was stirred for about 4 hours. 30 minutes before the solution was completely stirred, another solution was prepared by chelating TTBu with Acetylacetone (AcAc) with TTBu/AcAc molar ratio of 1/2. The latter solution was then stirred for about 30 minutes. As shown in Figure 3, both solutions were mixed together, and were then stirred for another 10 minutes before another EtOH was added into it with EtOH/TEOS molar ratio of 4/1. The resulted solution was stirred for another 1 hour.

Compositions (a) and (b) were then mixed together according to the weight ratio of 1 to 1. The mixed solution was stirred for 30 minutes and then left aging for another 3 days at room temperature. The resulted solution is known as VTT sol and was used as optical waveguide material.

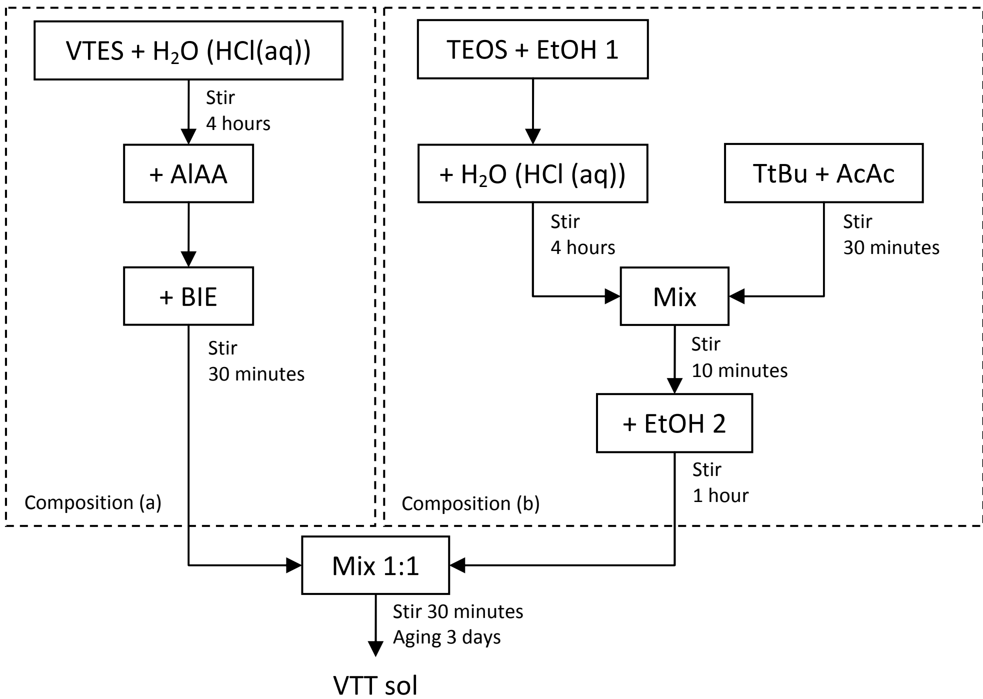

Figure 3 Preparation flow chart of VTT sol by sol-gel process

4.0 FABRICATION OF RIDGE OPTICAL WAVEGUIDE

VTT sol was deposited on quartz substrate by means of multi-step spin coating technique. Substrates were prepared in a size of 2 cm × 2 cm and cleaned multiple of times in the ultrasonic bath using deionized water with some detergent (Decon D90). They were then blown dried by means of high pressure filtered air and finally kept in the desiccator. The substrates were heated by rapid thermal processing (RTP) at the temperature of 300 °C prior to sol deposition.

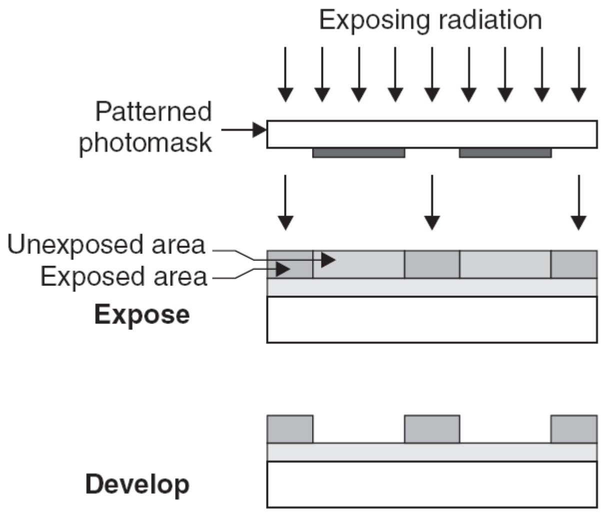

Figure 4 Direct photolithography and wet chemical etching process

Multiple layers of VTT sol coating were applied on quartz substrate in order to obtain a planar optical waveguide of suitable thickness. According to the work in [19], 3 layers of VTT sol spun at the speed of 1250 rpm were sufficient to get a thickness of about 3 μm. Each coating layer was baked at a temperature of 80 °C for 5 minutes in convection oven. The resulted 3 layers coating was then baked for another 10 minutes at the same temperature before left to be aged for 1 hour in the desiccator. The sample was then exposed to 250 mW/cm^2 of UV light irradiation through 4 μm width opening of a photomask for duration of 30 minutes in the presence of nitrogen gas flow such as illustrated in Figure 4. Ridge optical waveguide was then developed by means of wet chemical etching process. Etching solution consists of sodium hydroxide (NaOH), H_2O, and EtOH in the weight ratio of 5/15/25 was used. Finally, it was thermally cured using RTP at the temperature of 200 °C for about 60 seconds.

5.0 CHARACTERIZATION OF RIDGE OPTICAL WAVEGUIDE

The 2 cm × 2 cm ridge optical waveguide samples were naturally cleaved to two smaller experiment samples using diamond tip glass cutter. The cleaved facet was observed suitable for direct end-face fiber butt-coupling method. Ridge optical waveguide characterization experiments were conducted using equipments and facilities such as illustrated in

Figure 5. Light from a laser source at 1550 nm wavelength was coupled into a single mode fiber which had a spot size of about 8 μm. The light from the fiber was coupled directly toward the cleaved end-face of the ridge optical waveguides. Output light was collected using 20× microscope objective lens and directed towards an infrared imaging camera. The output beam profile was captured and analyzed using BeamView USB 4.4.2 beam analyzer.

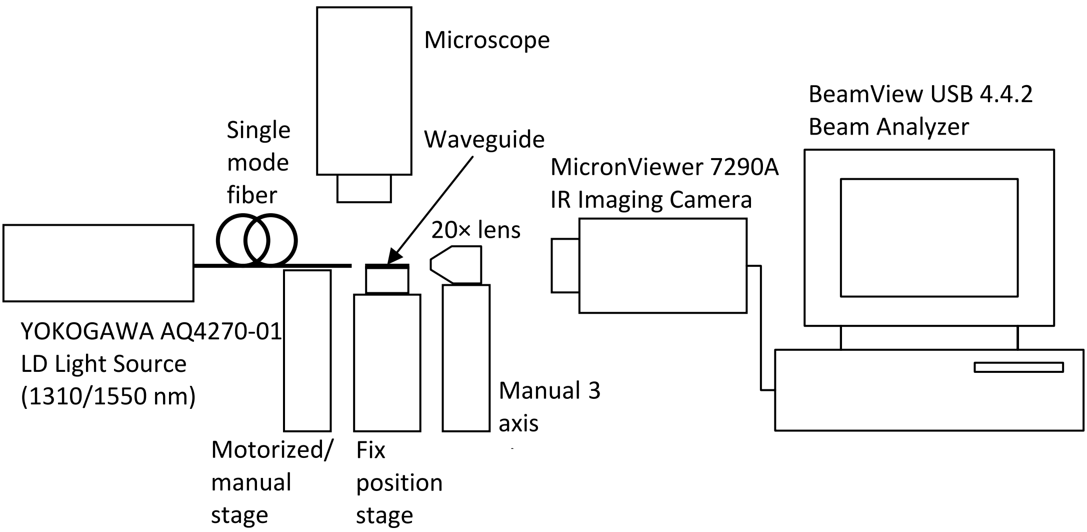

Figure 5 Equipments setup for ridge optical waveguide characterization

6.0 RESULTS AND DISCUSSION

Simulation on the general ridge optical waveguide structure was performed in order to roughly obtain a structure design that could guide the optical wave at 1550 nm wavelength. Figure 6 summarizes the result of the simulation. The structure width, *w* and thickness, *d* were varied from 2.0 to 7.5 μm and from 2.0 to 6.0 μm, respectively. The figure shows that the fundamental mode (mode 0) structure width cut-off condition was at about 2.5 μm regardless of changes in the structure thickness. However for the first mode (mode 1), it was observed to be inversely proportional to the structure thickness. In other word, a thicker structure need less width yet able to confine two propagation modes and vice versa. Shaded area in the figure shows the ranges of structure condition of which single mode ridge optical waveguide would be achievable.

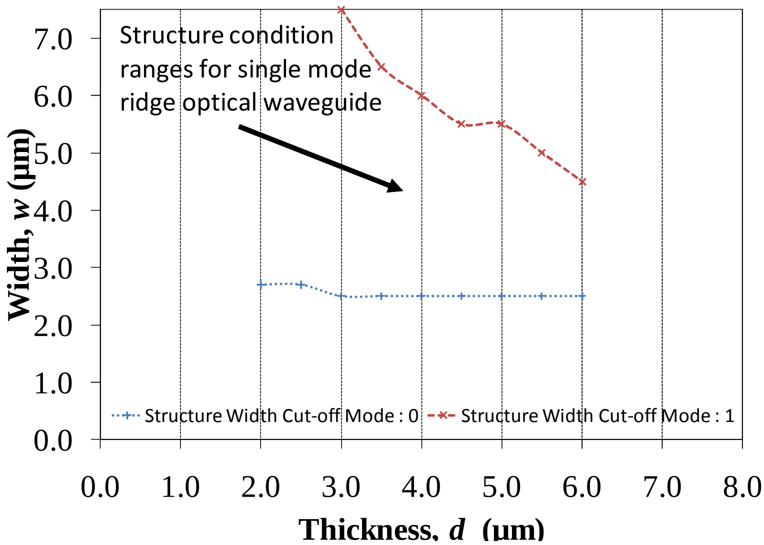

Figure 6 Structure cut-off condition for ridge optical waveguide

Direct end-face fiber butt-coupling of 1550 nm laser to the cleaved end-face of the ridge optical waveguide was conducted. Figure 7 shows the end-face of ridge optical waveguide fabricated with structure width and thickness of about 8 µm and 4 µm, respectively. Figure 8 shows the near-field output profiles of a 1550 nm laser from the end of the ridge optical waveguide. The pictures indicate that there were two propagation modes confined within the ridge optical waveguide fabricated. This result agrees well with the finding of simulation discussed previously. It proves that hybrid VTT sol-gel materials are able to guide optical wave and therefore are good candidates for optical waveguiding applications.

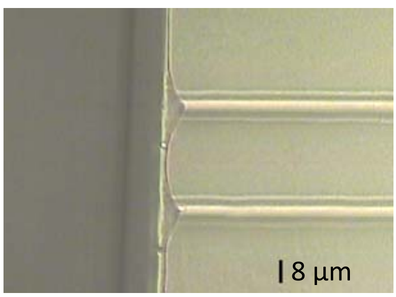

Figure 7 Microscopic picture of the end-face of ridge optical waveguide

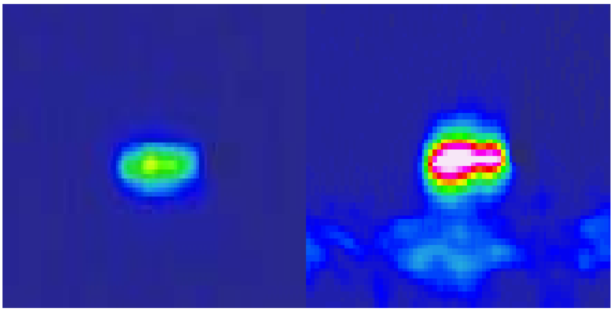

Figure 8 Near-field waveguide mode profiles of ridge optical waveguide

7.0 CONCLUSIONS

Ridge optical waveguide was designed using RSoft Photonic CAD Suite BeamPROP and fabricated using hybrid VTT sol-gel materials as waveguide core coating. Observation on the ridge optical waveguide output profiles of 1550 nm laser discovered that VTT sol-gel material showed the optical waveguiding ability. The number of modes confined within its structure of about 8 μm width and 4 μm thick are agreeable to the finding of simulation. It is also believed that a single mode ridge optical waveguide that is of practical use in many optical communication devices is realizable provided that the waveguide structure thickness and width are within certain range such as the finding in this work. Microscopic images of the end-face of the naturally cleaved ridge optical waveguide indicate a significantly acceptable quality. The ability to guide light at 1550 nm signifies that hybrid VTT sol-gel materials are good candidates for optical waveguiding application. Additional investigations and studies on the parameters such as propagation loss and thermooptic coefficient of the proposed VTT sol-gel materials will be embarked to further convince the materials' suitability for active optical communication devices development.

ACKNOWLEDGEMENTS

This work is funded by MOSTI-Escience grant no. 01-01-06-SF0656. Fabrication facilities and characterization equipments are provided by AMREC SIRIM Bhd., Telekom Malaysia Innovation Centre and Photonics Research Centre, University Malaya.

REFERENCES

[1] R. Gvishi, 2009. Fast Sol-Gel Technology: From Fabrication to Applications. *Journal of Sol Gel Science and Technology*. 50: 241-253.

[2] P. Coudray, P. Etienne, and Y. Moreau, 2000. Integrated Optics Based On Organo-Mineral Materials. *Materials Science in Semiconductor Processing*. 3: 331-337.

[3] J. Zarzycki, 1997. Past and Present of Sol-Gel Science and Technology. *Journal of Sol Gel Science and Technology*. 8: 17-22,

[4] X. Zhang, M. Qian, X. Zeng, Z. Zhao, J. Lasante, and P. Plante, 2008. Design and Fabrication of Single Mode Rib Waveguides Using Sol-Gel Derived Organic-Inorganic Hybrid Materials. *Journal of Sol Gel Science and Technology*. 45:103-107.

[5] G. C. Righini and S. Pelli, 1997. Sol-Gel Glass Waveguides. *Journal of Sol Gel Science and Technology*. 991-997.

[6] H. Schmidt, 1984.Organically Modified Silicates by the Sol-Gel Process. *Mater. Res. Soc. Symp. Proc*. 32: 327-335.

[7] H. Scholze, 1985. New Possibilities for Variation of Glass Structure. *Journal of Non Crystalline Solids*. 73: 669-680.

[8] F. Del Monte, P. Cheben, C. P. Grover, and J. D. Mackenzie, 1999. Preparation and Optical Characterization of Thick-Film Zirconia and Titania Ormosils. *Journal of Sol Gel Science and Technology*. 15: 73-85.

[9] W. Que, Z. Sun, Y. L. Lam, Y. C. Chan, and C. H. Kam, 2001. Effects of Titanium Content on Properties of Sol-Gel Silica-Titania Films Via Organically Modified Silane Precursors. *Journal of Physics D: Applied Physics*. 34: 471-476.

[10] W. Que, Y. Zhou, Y. L. Lam, Y. C. Chan, H. T. Tan, T. H. Tan, and C. H. Kam, 2000. Sol-Gel Processed Silica/titania/y-glycidoxypropyltrimethoxysilane Composite Materials For Photonic Applications. *Journal of Electronic Materials*. 29: 1052-1058.

[11] B. Wang and L. Hu, 2005. Effect of processing parameters on the optical properties of TiO_2/ormosil planar waveguide. *Journal of Sol-Gel Science and Technology*. 34: 71-76.

[12] M. Zaharescu, M. Crisan, L. Predoana, M. Gartner, D. Cristea, S. Degeratu, and E. Manea, 2004. Hybrid Inorganic-Organic Sol-Gel Coatings in the SiO_2-TiO_2 System. *Journal of Sol Gel Science and Technology*. 32: 173-177.

[13] P. Innocenzi, A. Martucci, M. Guglielmi, L. Armelao, S. Pelli, G. C. Righini, and G. C. Battaglin, 1999. Optical and Surface Properties of Inorganic and Hybrid Organic-Inorganic Silica-Titania Sol-Gel Planar Waveguides. *Journal of Non Crystalline Solids*. 259: 182-190.

[14] M. Yoshida and P. N. Prasad, 1996. Sol-gel-processed SiO_2/TiO_2/poly(vinylpyrrolidone) Composite Materials for Optical Waveguides. *Chemistry of Materials*. 8: 235-241.

[15] K. Tadanaga, K. Ueyama, T. Sueki, A. Matsuda, and T. Minami, 2003. Micropatterning of Inorganic-Organic Hybrid Coating Films from Various Tri-Functional Silicon Alkoxides with a Double Bond in Their Organic Components. *Journal of Sol Gel Science and Technology*. 26: 431-434.

[16] C. Y. Jia, W. Que, and W. G. Liu, 2009. Preparation and Optical Properties of Sol-Gel Derived Photo-Patternable Organic-Inorganic Hybrid Films for Optical Waveguide Applications. *Thin Solid Films*. 518: 290-294

[17] X. Luo, C. Zha, and B. Luther-Davies, 2004. Anhydrous Sol-gel Synthesis of Titania-doped Siloxane Polymer for Integrated Optics. *Journal of Sol Gel Science and Technology*. 32: 297-301.

[18] K. Tadanaga, T. Sueki, A. Matsuda, T. Minami, and M. Tatsumisago, 2006. Micropatterning of Inorganic-organic Hybrid Thick Films from Vinyltriethoxysilane. *Nippon Seramikkusu Kyokai Gakujutsu Ronbunshi/Journal of the Ceramic Society of Japan*.114: 125-127.

[19] A. S. Abdullah, N. M. Kassim, M. H. Ibrahim, M. Z. A. Malek, and A. Nawabjan, 2010. Fabrication and Characterization of Planar Optical Waveguides Based on Multi-Precursor Vinyltriethoxysilane/Tetraethoxysilane/Tetrabutoxytitanate Organic-Inorganic Sol-Gel Derived Materials," in *International Conference on Experimental Mechanics (ICEM2010)* Kuala Lumpur.