

Design and Fabrication of an Ion Exchange System

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Abstract: *In this work, the design, fabrication and optimization of a novel ion exchange unit are presented. A simple low cost system with a modified dipping mechanism and temperature controller for ion exchange in the glass substrate with molten salt is described. The design and technical details of the system together with those of the temperature controller are discussed. The ion exchange unit is optimized with Ag^+/Na^+ ion exchange in commercially available soda lime glass. The energy dispersive spectrum and absorption spectrum of the ion exchanged glass are discussed. The system is found particularly suitable for exchange of ions in the glass for the planar waveguide fabrication and also suitable for thin film fabrication by dip coating method.*

Keywords: Ion Exchanger, Waveguide, Ag^+/Na^+ ion exchange, ATmega32 microcontroller, Surface Plasmon Resonance

1. Introduction

Ion exchange is one of the most prevalent, generic and effectual treatment method for waveguide fabrication, glass strengthening and conductivity enhancing [1]-[3]. Ion exchange is a process in which mobile ions from an external melted salt are exchanged with electrostatically bounded ions in the functional groups contained within a solid glassy matrix. It is a well developed technique, and has been employed for many years in the areas of research in glassy materials. In spite of this advanced stage of development, various aspects of ion exchange technology are being studied to improve its competence and economy in its application to waveguide fabrication. The efficiency of the ion exchange process mainly depends on the thermal equilibrium of the melted salt, dip and withdrawal speed of the substrate and dwell duration of the substrate in the melted salt. Even though there are various methods developed for ion exchange in different field of applications, here we report the design and fabrication of a novel ion exchange unit with the above temperament for waveguide applications. Ion exchange in glass is a well established method for the fabrication of passive and active integrated photonic devices [4], [5]. By a relatively simple fabrication process, it is possible to develop ion exchanged waveguides for active and passive devices with very low propagation losses, excellent mode matching to optical fibre and low waveguide birefringence [6]. For waveguide lasers and amplifiers, the ion exchange process is superior due to its compatibility with glass substrates having high rare earth ion concentrations. Ion exchange is an engineering process and was originally used to improve the surface mechanical properties of glass substrate. In which the cations in glass (Na^+ , K^+ , Li^+) are exchanged with other cations having the same valence (Ag^+ , Rb^+ , Cs^+ , Li^+ , K^+ , Tl^+) [5], [6]. If ions of high refractive index modifying ability are replaced by those of low refractive index modifying ability, eg. Ag^+ by Na^+ , Li^+ by Na^+ , etc. the eventual glass has a gradient in the refractive index which decreases from the center towards the periphery. The process is diffusion driven, either thermal diffusion or electric field assisted ion migration. Ion exchange in glass produces a graded refractive index profile. This method has enabled inexpensive fabrication of graded index (GRIN) lenses that are utilized as imaging lenses in small photocopiers and scanners [7], in endoscopes enabling minimally invasive surgery [8], and in collimation optics

used in high power diode lasers [9], [10]. Ion exchange can also be used to fabricate low loss channel waveguides for planar lightwave circuits (PLCs) in glass substrates.

We have designed and developed an ion exchange system with programmable up and down movement mechanism of the sample. The ion exchanger consists of a dipping mechanism and a temperature controller. The dipping can be controlled using microcontroller via electronic control circuit. We have successfully employed the set up for the fabrication of optical planar waveguides via Na^+/Ag^+ ion exchange in commercially available soda lime glass. Here we report the architecture, electronic circuit description, performance and optimization of the ion exchange system. The feature of our system is its convenience to exchange the cations bounded in glass by any other cations of same valency by advisable selection of melt composition in the inside chamber. Since thermal equilibrium is one of the main criteria for the ion exchange process, the temperature control should be done very precisely. Removable substrate holder and visibility of the movement of substrate are other admirable features of this system.

2. The Ion Exchanger Architecture

The system fabricated here is a modified version of dip coating setup. The usual dip coating system works on several up and down movement of the substrate to be coated. But in the ion exchange system the salient transfiguration is precise up-down movement. In which the substrate movement mechanism gives consent for a very slow dipping speed of 2 $\mu\text{m/s}$. This lesser speed aids the immersion of glass substrate without any fracture due to thermal shock.

Fig. 1 shows the schematic of the ion exchange system. The important parts of the system are programmable translation unit, mechanical setup consisting of a substrate holder, inner chamber with heater coil, an enclosed outer chamber and digital temperature controller. The translational unit consists of a programmable microcontroller based stepper motor control drive for translation of the substrate holder. The translational rate can be controlled by varying the speed 2 - 9000 $\mu\text{m/s}$. In this setup, linear translation is accomplished by directly coupling the motor shaft with a metal rod arrangement for ultra-smooth vibration free up and down movements. The number of dipping and the length of

dipping can be controlled programmably. The maximum translational length was 240 mm. Dipping length specifies how far the substrate should be moved downwards for a dip and it is varied from 0 to 240 mm.

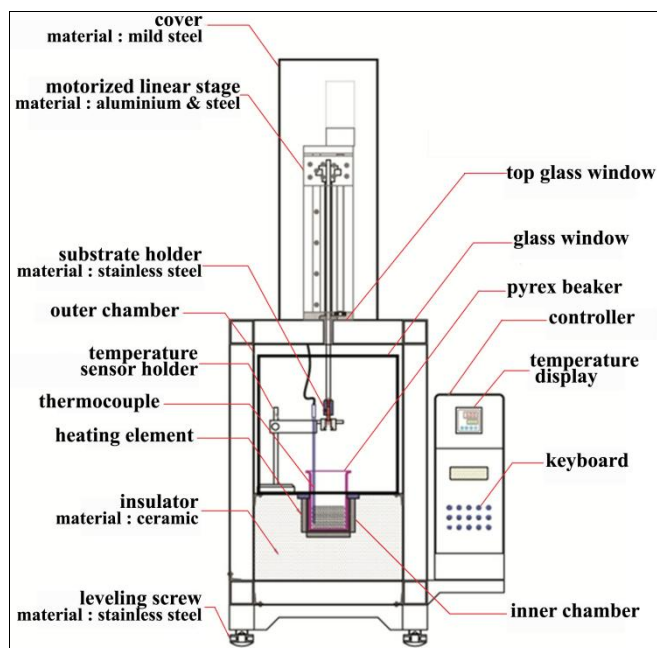


Figure 1: The ion exchange system

Mechanical setup consists of removable substrate holder and can be fitted to the end of the metal rod. Depends on the size and shape of the substrate used, the substrate holder can be changed. Ion exchanging can be done either vertically or horizontally by placing the substrate either X or Y plane using different types of substrate holders.

The inner chamber is made up of stainless steel with 90 mm depth and 85 mm diameter. This chamber acts as a top loading furnace in which a beaker can be slowly introduced. The height is limited to 90 mm so that the Pyrex beaker (of 115 mm height) can be placed by hand while it is being put into the chamber. Its top has a circular opening in order to insert the beaker. It has heating elements (nichrome coil) around its walls and also at the bottom. This helps to provide a uniform heating inside the chamber. The temperature of the heater can be controlled by a digital temperature controller to which a thermocouple is attached. The temperature of the inner chamber can be controlled in the range 30 to 500°C with an accuracy of 1°C.

There is also an outer chamber enclosing the inner chamber, translational unit and a thermocouple. This outer chamber also prevents the heat radiation. This chamber has a front door with a glass window to see the movement of the substrate while dipping. Visibility of the beaker contains melt makes it possible to ensure proper melt conditions and also to remove any impurities present before introducing the sample. There is another glass window on the top side of the outer chamber, to see whether the substrate is completely dipped. After the temperature is risen to the desired level, the user can start the dip process. The dip speed and the dwell time can be controlled by microcontroller.

3. Electronic Circuit Description

Fig. 2 shows the block diagram of the controller circuit cum dipping mechanism. The core of the system is prepared with an Atmel ATMEGA-32 microcontroller. It is a low-power CMOS 8-bit microcontroller with 40 pins and is operating at 16 MHz clock frequency. It has three linear and regular main memory spaces for data storage, which are the data memory, the program memory space and an EEPROM memory. The EEPROM has a tolerance of at least 100,000 write/erase cycles. It can hold 1024 bytes of data and is established as a separate data space. The microcontroller ATMEGA32 has a 10 bit A/D converter, internal PWM channels, UART/USART and there are up to eight inputs of 0-5 V available on port A. The pin driver is cogent enough to drive LED displays without procrastinate. The I/O ports are bi-directional. The digital parallel ports of the microcontroller serve as an interface amid different components. The ATMEGA-32 interfaces the LCD output display, keyboard input and driver card.

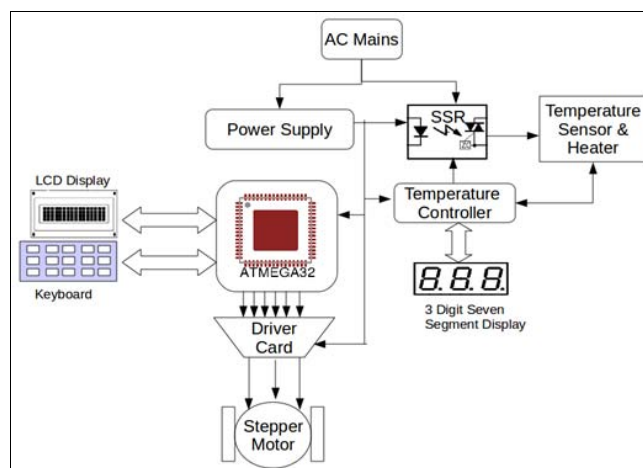


Figure 2: Block diagram of the temperature controller cum dipping mechanism

IC TMC249 with 28 pin configuration is utilized as the driver card. The driver IC TMC249 drives the current from the microcontroller to the stepper motor. It helps to buffer and drive the stepper motor in microcontroller. This IC is a dual full bridge driver for bipolar stepper motor control purposes. It supports up to a current of 4000 mA. Current control characteristics of the driver card permits to cool motor and driver operation. The output signal of the microcontroller is amplified to the level by TMC249 chip could rotate the stepper motor and regulate its speed. The stepper motor is an electrochemical appliance which renders current pulse into motor rotation. The speed of the motor can be regulated by altering the interval of the timer interrupt and the rotation angle by numerating the number of steps. Rotational speed of the motor shafts is directly related to the frequency of the input pulses and the numeral of input pulses decides the length of rotation. From the port of the microcontroller, input is given to the circuit which is a TTL logic. The output of the circuit is fed to the stepper motor's winding. Fig. 3 represents the speed detection process of the stepper motor. A photosensor VTR17D1H is used to detect the rpm and speed of stepper motor and a builder circuit to detect the signal received by photosensor. A reflecting surface placed on the motor detects the position and rpm of

stepper motor by the reflection of IR rays emitted from the diode. For encountering reflecting surface, a sine wave of 2V is obtained. This is converted into a square wave for the functioning of microcontrollers timer interrupt to calculate the rpm position.

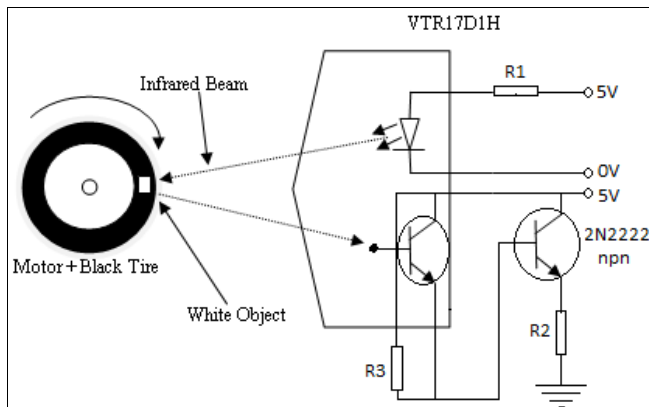


Figure 3: Speed detection of stepper motor

The temperature controller used is PID controller TC513 (SELEC India) with a temperature accuracy of $\pm 1^\circ\text{C}$. This temperature controller unit can display and accomplish the temperature up to a maximum value of about 500°C . The required temperature can be set depending upon the melting point of the salt in the beaker. The thermocouple used here is the K-type (Chromel-Alumel type), which can be effectively utilized to sense the temperature in the range $250\text{-}500^\circ\text{C}$. The output of the thermocouple is calibrated to $41\ \mu\text{V}/^\circ\text{C}$. A 230 V main supply is fed to the Photocoupled solid state relay (SSR) and power supply unit (PSU).

The power supply unit (PSU) supplies sufficient dc voltages of 5 V, 12 V, 24 V for different circuit boards. The 12 V dc power is supplied to exhaust fan to cool down the electronic circuit board. The PSU provides 5 V power to the microcontroller, LCD and keyboard. The power supply unit also provides voltage for the optocoupler inside the SSR to function. Microcontroller produces 5 V pulse signals according to the data entered through the keyboard (input). The stepper motor is designed to work at 24 V. These 5 V pulse signal and the 24 V power supply is fed to the driver controller and it drives the stepper motor according to the 5 V pulse signal from the microcontroller. Fig. 4 displays the interface for the ATmega32 programming. Resistors for all pin coursing to the parallel ports are invulnerable resistors and they conserve the parallel port by confining the current. LEDs before the regulators and after the regulators used to guard from voltage fluctuations. LED for RESET port indicates RESET has been applied or not. The capacitors

parallel with the voltage regulator are used to attenuate noise and stabilize voltage. The external clock is utilized to enhance the speed. After constituting the programmer, some software is needed to compile the C code. A hex file should be necessary and it burns onto the microcontroller. The complications for writing code in different operating systems are eschewed here by using GCC as the compiler. The utilities required such as avr-gcc, avr-libc, avr-binutils and avrdude are included in WinAVR (Integrated Developed Environment for Atmel products). A program file (ending in .c) and a makefile (generated using makefile generator) are imperative in WinAVR. A programmer's notepad is utilized to compile and burn the program onto the microcontroller. In the makefile generator, selected the apposite options and accorded the C source file. Source file and makefile are saved in the same directory. For compilation picked "Tools→ [WINAVR] Make All" and checked for errors, then chose "Tools→ [WINAVR] Program" for burning the code onto the microcontroller.

There are seven parameters that should enter through the keyboard of the microcontroller for regulating the speed, dip length and dwell duration of the substrate holder. The following are the parameters. 1. Start position – It indicates the initial position of the substrate holder. It can alter from 0 to 240 mm. 2. Dip length – It denotes how far the substrate holder moves from the start position. 3. Dip speed – It signifies the speed of the substrate holder from start position. Its value should be in $\mu\text{m/s}$ or mm/s . It changes from $2\ \mu\text{m/s}$ to a maximum of $9\ \text{mm/s}$. 4. Dip duration – Dip duration is the time during which the substrate holder exists in the desirable depth. It can emphasize in s/min/hr . 5. Return speed – It is the speed at which the substrate holder returns back to its original position. It can also represent in $\mu\text{m/s}$ or mm/s . 6. Dry duration – It is the time at which the substrate tarries at the start position after returns. It can propound in s/min/hr . 7. Number of dips – It implies the number of times the substrate holder dips in the melt. Before the microcontroller performance the substrate holder may be on any position within the maximum vertical length of 240 mm as mentioned earlier. When we execute the microcontroller program, the substrate holder rapidly moves to the start position. When the program runs by entering the parameters, corresponding signals are given to the driver card. The stepper motor functions in accordance with it. The control parameters after the control signals of driving the motor can be input through the 3×5 matrix membrane keyboard connected to the microcontroller. The 4×20 characters LCD panel displays the parameter information.

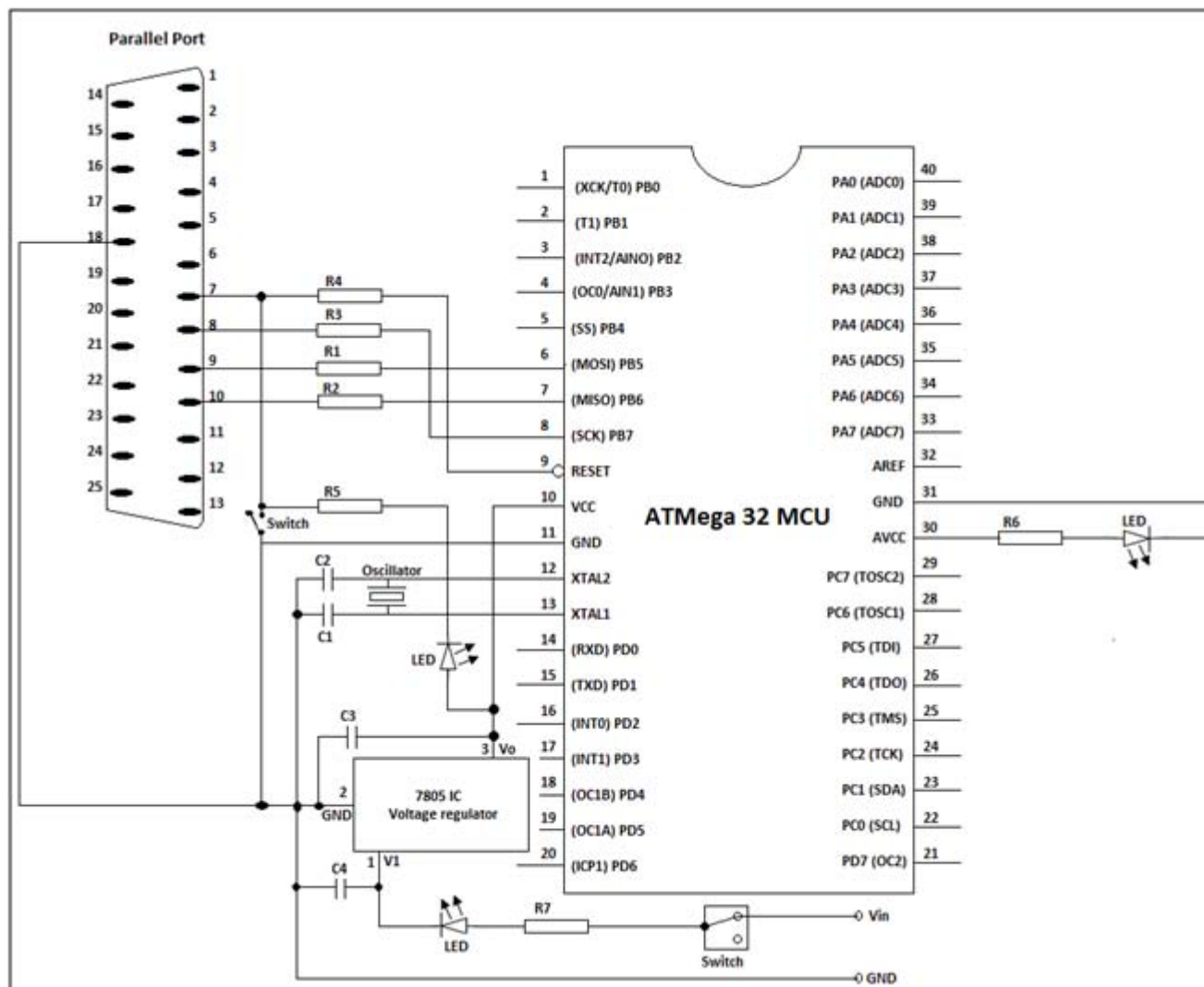


Figure 4: ATmega32 programming interface

4. Performance

The substrate is inserted between the plates of the substrate holder and the knob is fastened to hold it firm. Proper composition of chemicals to be melted is taken in a pyrex beaker and is inserted in the inner chamber. Using the temperature controller keys, temperature is set to a predefined value and waits until it reaches the same. Hence the sample is sandwiched between the plates which are an extension of suspension mechanism, through which the sample is slowly immersed into the molten salt (the sample is very slowly introduced into the beaker and is kept just above the melt to attain a thermal equilibrium and can avoid thermal shocks). The immersed sample remains there for a certain amount of time. After that it is withdrawn from the salt and held above the molten salt to avoid the thermal shocks. The above translation of substrate is controlled programmably by parameters entered through the keyboard which is connected to the microcontroller. By entering the dip start position and dipping length, the substrate can be move from 0 to 240 mm along the vertical direction. For example, if the dip start position is 40 and the dipping length is 120, the substrate reaches 40 mm from the reference point and it travels 120 mm along the vertical direction. Dip duration can be set, which specifies how long the substrate should remain dipped in the solution. The sample is then

transferred into a beaker containing deionised water in order to wash the salt residues completely and make the surface clean. Finally the exchanged surface is cleaned up with distilled water and acetone using soft and lint-free Kodak lens paper.

5. Optimization with Ag⁺/Na⁺ ion Exchange

The silver ions are introduced into the glass network by an isothermal ion exchange process for the optimization of the ion exchange system. Slices of commercially available soda lime glasses are used for the exchange of ions, by keeping it in a molten salt with a composition of 5% AgNO₃ and 95% NaNO₃.

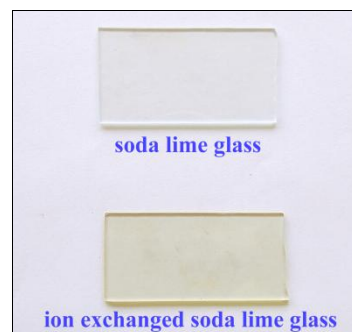
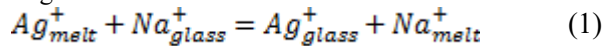


Figure 5: Photographs of the soda lime glasses

Fig. 5 shows the photographs of the ion exchanged soda lime glass along with the substrate glass. It is noted that color of the soda lime glass becomes yellowish due to the Ag^+/Na^+ ion exchange in it. Ion exchange in soda lime glass has been carried out for 10 seconds duration at $400^\circ C$. The dipping and withdrawal speed of the substrate was $150 \mu m/s$. The parameters like temperature, dip duration, dipping and returning speed for the ion exchange process were optimized by trial and error method to get the crack free ion exchanged glass. The ion exchange process can be represented by the following chemical reaction.



where subscripts „melt“ and „glass“ represent the cations in the melted salt and in the glass substrate, respectively.

The soda lime glass used here contains sodium, magnesium, aluminum, silicon and calcium, and it is verified by energy dispersive spectrum (EDS) analysis. Fig. 6 shows the EDS spectrum of ion exchanged soda lime glass. Appearance of peak at 3.1 keV in the EDS spectrum reveals the presence of Ag^+ ions in the ion exchanged glass [11]. Table 1 shows the composition of ion exchanged glass which is obtained from the EDS spectrum. It reveals that 1.85 wt% of Na^+ ions are replaced by Ag^+ ions due to the ion exchange process.

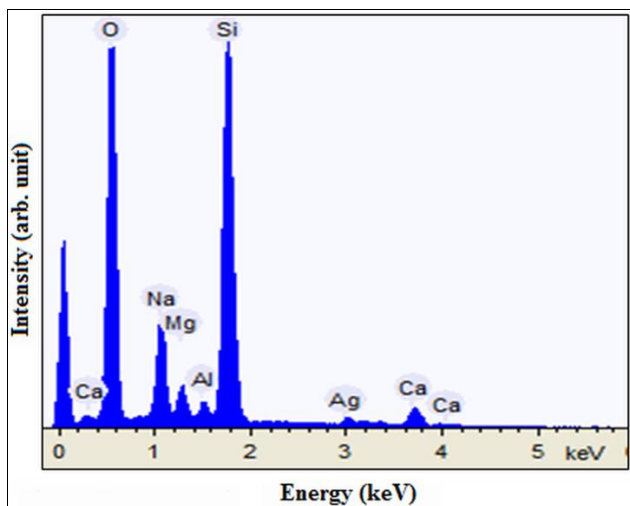


Figure 6: EDS spectrum of ion exchanged soda lime glass for 10 seconds at $400^\circ C$

Table 1: Composition of ion exchanged glass obtained from EDS spectrum

Element	Weight %	Compound %	Formula
Sodium	10.490	14.140	Na_2O
Magnesium	2.997	4.969	MgO
Aluminum	1.594	3.012	Al_2O_3
Silicon	33.331	71.305	SiO_2
Calcium	3.277	4.585	CaO
Silver	1.853	1.990	Ag_2O
Oxygen	46.459	-	-

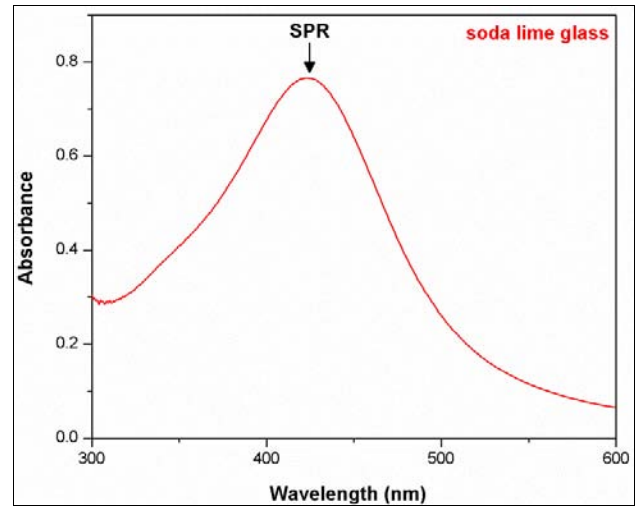


Figure 7: Absorption spectrum of ion exchanged glass

Fig. 7 shows the UV-Vis absorption spectrum of an ion exchanged soda lime glass annealed for 5 hours at a temperature of $450^\circ C$. After the annealing process, the sample color changes to pale red [12]. Surface plasmon resonance (SPR) band with a maximum of 422 nm in the absorption spectrum indicates the presence of silver nanoparticles (NPs) in the ion exchanged glass substrate due to the annealing process [12]-[15]. Diameter (d) of the silver NPs can be estimated using the equation [16], [17].

$$d = (2hv_f / \Delta E_{1/2}) \quad (2)$$

where $\Delta E_{1/2}$ is the full width at half maximum of SPR band (in eV), v_f (1.39×10^6 m/s) is the Fermi velocity of electrons in bulk silver and h is Planck's constant (in eV s). For ion exchanged soda lime glass, the values for $\Delta E_{1/2}$ and the particle size of Ag NPs were obtained as 0.69 eV and 17 nm, respectively.

6. Conclusions

Novel ion exchange system with a temperature controller is designed, fabricated and optimized. We have successfully fabricated optical planar waveguides by Na^+/Ag^+ ion exchange in soda lime glass. The EDS spectrum of the ion exchanged soda lime glass revealed that Na^+-Ag^+ ions are exchanged between melt and glass. From UV-Vis absorption spectrum, it is evident that Ag nanoparticles were formed by annealing the Na^+-Ag^+ ion exchanged soda lime glass at $450^\circ C$ for 5 hours. To sum up it can be stated that the present system can be employed for ion exchange and dip coating through which one can produce waveguides and thin films, respectively.

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