Glasses for ion-exchange technology

F. Rehouma and K. E. Aiadi

***Abstract*—** Integrated ion exchanged devices on glass seems to be as one of the most promising technologies for the development of optoelectronic field. That is due to the potentiality of glass material, facility and flexibility of the fabrication process, the cost of the elaborated devices and the high performances of these components. In this paper, a review on glasses used for ion exchanged structures is given. The structure and the influence of the composition on the glass properties are presented. The ion exchange technology is described. The relation between the glass compositions and the exchanged ions is treated. Some compositions of glasses suited to this technology and the characteristics of elaborated structures are given. Finally selection criteria of glasses adapted to this technology are proposed.

***Keywords*—** integrated optical waveguide, ion-exchange, optical sensors, propagation losses, refractive index, silicate glass.

I. INTRODUCTION

Ion exchange technique is well known for more than a century ago. It has been used to produce tinted glass and improve its mechanical properties. It is observed that ion exchange was also producing an increase of the refractive index in the diffused layer. Since 1972 the last property was explored in order to realize integrated optical waveguides on glass substrates [1] [2] [3] [32]. Now, glass waveguides are considered to be prime candidates for optical signal processing applications in optical communications, sensors, and other related areas. This technique is simple, and does not require sophisticated equipments. The glass waveguide components fabricated by ion exchange process is characterized by compatibility with optical fibers, potentially low cost, low propagation losses and the ease of their integration into the system. Recently, the flexibility of this technique has been demonstrated. Such flexibility permits the obtaining of an integrated waveguide with different depths a long of glass substrate [4] [5] [30] [31]. These performances and qualities depend mostly on the glass substrate composition and the exchanged ion (dopant). The glass used in this technology must have some conditions to fulfill. It must contain enough of alkaline in order to create an

important variation of refractive index; low intrinsic losses; the refractive index of the material is close to that of the silicon; chemical durability; no bubbles and high degree of homogeneity; easy diffusion of the ions; simple production; the fusion temperature is higher than the diffusion temperature; and finally, a low Haven ratio.

In this paper, we present a review on glasses used for ion exchange technology, which satisfied the conditions quoted above. The review covers also the specified ions used for each glass, the main features of the process, the influence of materials on the fabrication conditions and the integrated compound performances. The paper is organized as follow. In the first part we present the general structure of the glass adapted to the ion exchange technology. The influence of the glass composition on the glass characteristics and the fabrication conditions is treated in the same part. The second part is reserved to the ion exchange process. In this section, we describe the principal of ion exchange; the fabrication parameters; the influence of glass composition on these parameters; and the performance of the fabricated compounds related to the kind of the used glass. Special glasses developed for specific ion-exchanged ions are discussed in the third part. We finish the paper by a discussion about future development of glasses in order to ameliorate the qualities and the fabrication conditions of realized waveguide.

II. GLASS MATERIAL

The glass is a familiar product. Its internal structure remains unknown. Amorphous materials do not possess periodicity at large scale. A glass composition is so complex that it is extremely difficult to predict by mathematical simulation the actual properties of a given glass. However the long experience of the glass makers gives them the ability to have a good idea, at least for the most classical compositions, of what will be the properties and how they will change with small modifications of a given constituent. This is part of the "art" of glass making.

A. Glass structure

The oxides in general formula of A_mO_n constitute the majority of optical glasses. Several models are proposed in order to describe the glass structure [6]. These models take the ZACHARIASEN one as a support [7], who classifies the oxides in three categories:

-The basic building blocks of oxide glasses are SiO_2, B_2O_3, GeO_2, P_2O_5, and As_2O_3 tetrahedral. These oxides with strong bonds are called network formers. The network former can form glasses by themselves without addition another oxide, the SiO_2 as an example. Their structure is generally constituted by disorderly chains of triangles (A_2O_3

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and AO2) or tetrahedral (A2O5). Glasses with large relative amounts of network formers have higher glass transition temperatures and, in general, higher viscosities at all temperatures;

- Glass strength can be enhanced by introducing into the glass matrix so called network intermediates, such as ZnO, Al2O5, CdO, TiO2 and PbO. Though network intermediates cannot form glass matrix by themselves, they contribute to the glass strength;

- Finally, glasses can be tailored to have desired properties by adding network modifiers, such as Na2O, Li2O, K2O, CaO, MgO, SrO, and BaO. These characterized by relatively loose bonds to the remainder of the network.

B. Glass properties

Glass properties can be classified into two categories, according to its linear variation or not with the glass composition. In the first case, empirical models are used to obtain the variations of characteristic parameters of glass, such as the density, refractive index, molar volume…

In the second case, it is delicate to calculate accurate values. Only some tendencies can be given. The concerned parameters are those linked to the ionic mobility as conductivity, diffusion's constants and electric mobility or the mechanical and thermal properties of glass.

Some glass properties are important for ion exchange waveguide fabrication. Among those; the refractive index is defined as ratio of the light speed in the vacuum and light speed in material medium. This parameter is related to the composition of glass oxide by an empirical relation [8]. The relation gives values for the refractive index very close to the measured ones. The introduction of alkaline oxides in silicate glasses increases the refractive index. The same tendency if we add the oxides CaO, MgO, ZnO, PbO, B2O3. The Al2O3 with small quantity (Al2O3/alkaline oxide less than 1) in glass leads to a small rising in refractive index. If the ratio is more than 1, in this case Al2O3 is as a former and it causes an important elevation of refractive index. The Fluorine in the glass decrease the refractive index but its quantity is limited to less than 4%. More than this quantity, the glass become delicate and the chemical durability decreases.

The second important parameter is the ionic conductivity. In oxide glasses, only the modifier ions have a loose bond, which permit it to move under a temperature of few hundred of degrees Celsius. The conductivity can be measured easily by impedance's spectroscopy method. The introduction of alkaline oxides as modifier network, Al2O3 and the nitrogen increases the conductivity. On the contrary, CaO, MgO, ZnO, PbO, B2O3, reduce the conductivity [9];

The chemical durability is the third important parameter for glasses. It is defined as the chemical resistance to the acids and other degradation agents. In glass, the bond Si-O can be damaged with the attacks of HF acid. The speed of damage depends on the pH of HF solution. However, the presence of CaO, MgO, ZnO, PbO, B2O3 and Al2O3 [10] increase the chemical durability of glass;

The oxide glasses are characterized by a high transparency in the visible and near- infrared. The existence of impurities in glass as transition elements (Cr, Mn, Fe, Co, Ni, and Cu) causes some absorption in the visible and near infrared bands.

III. Ion exchange in glass

If Planar integration is vitally important in the vision of all-optical networks. Integration of complex functions will reduce costs in the long run, and allows for the manufacture of multiple circuits on a single wafer [11]. Ion exchange offers a relatively simple method of integrated waveguide manufacturing and is far superior to the deposition process [12]. The major advantages of the technique are:

- Possibility to obtain passive or active devices;
- Very low losses and compactness of compounds;
- Mass production which leads to the reduction of the device costs.

The principle of the technique is simple in which an ion (A^+) associated with a solid glass matrix (X^-) is replaced by another ion (B^+) with the same valence in melt salt:

$$X^-A^+ + B^+ \rightleftarrows X^-B^+ + A^+.$$

The (A^+) represent the alkali oxides that compose the glass (Na^+, K^+, Li^+) and the ion (B^+) monovalent cations in the molten salt. The monovalent cations can be exchanged such as $Ag^+, K^+, Cs^+, Li^+, Tl^+$ and Rb^+ [16]. Recently Cu^+-Na^+ exchange has also been reported [17]. This exchange modifies locally the chemical composition of the surface glass fig. (1). This variation due to the diffusion of the ions of the two species causes a local elevation of the refractive index in the diffused zone leading to the creation of a guiding structure. This change occurs due to the combination of atomic size and polarisability of the exchanging ions [12]. In the table 1 the polarisabilities and radius of different ions available for ion exchange in glass is given.

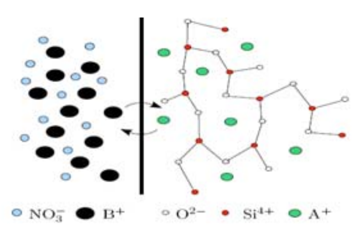


Table I. Ions available for exchanged in glass.nonlinear data to a higher dimensional feature space

Ions	*polarisability* ($\mathring{A}^3$)	*radius* ($\mathring{A}$)
Lithium (Li)	*0,03*	*0,65*
Sodium (Na)	*0,43*	*0,95*
Silver (Ag)	*2,4*	*1,26*
Potassium (K)	*1,33*	*1,33*
Rubidium (Rb)	*1,98*	*1,49*
Thallium (Tl)	*5,2*	*1,49*
Cesium (Cs)	*3,34*	*1,65*

If the glass is covered by a thin film impermeable to ion exchange in which a window has been etched by a standard photolithographic process like in microelectronics, a channel waveguide is elaborated. Fig. (2) shows the main steps for manufacturing a channel waveguide in a glass wafer. A plate of optical glass is well polished and carefully cleaned. Onto it a thin aluminum layer is evaporated in a high vacuum chamber. Then, using the standard technique of photolithography one can transfer the mask of the device onto the metal layer. The plate is spincoated with photoresist and exposed to UV light through a mask which supports the pattern to be transferred [13][14]. After developing the photoresist and etching the metal film in appropriate baths, the mask pattern is transferred to the aluminum film and the smallest open windows obtained have a width of about 2 μm.

The next step is the ion-exchange process performed for example, in a pure potassium nitrate molten bath (KNO_3) or mixture of sodium nitrate and silver nitrate $(NaNO_3 + AgNO_3)$ at a temperature between 330 and 450 ^0C. Producing a low-loss single-mode straight waveguide at a given wavelength, the diffusion parameters (temperature, exchange time, width of the open windows) should be well-chosen. Then potassium or silver ions diffuse into the glass where the aluminum film have been eliminated, and replaced by sodium ions and it results in a maximum refractive index increase near the surface. A final step involves a second exchange with applying an electric field in order to bury waveguides under the surface of the substrate [15] [33]. Burying the waveguides is necessary to reduce the propagation losses and efficient fiber coupling.

Many ion-exchange processes have been developed using different ions. The table 2 summaries the characteristics of the different exchanged ions in terms of propagation losses, refractive index variation, conditions of diffusion etc…

The ion-exchange technique is now well-known for the realization of single-mode waveguides in the wavelength ranges of 0, 6-0, 8 μm for optical sensors applications and 1.3-1.55 μm for telecommunication applications.

Since 1972, many works have been published. Different ions available for ion exchange are tested on different glasses. They show that the fabrication conditions and the waveguide qualities depend directly on the exchanged ion and the glass composition. The Table 3 summarizes some of those works.

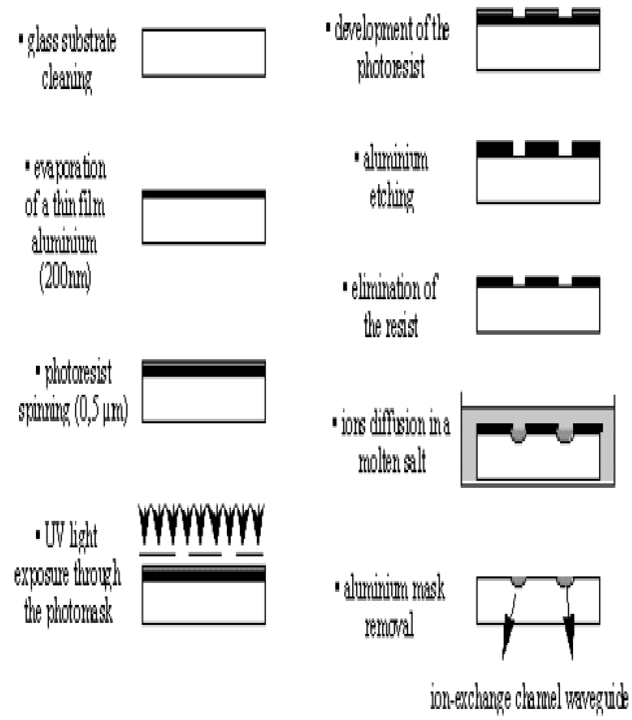


Fig.2 The main steps for manufacturing a channel waveguide in a glass substrate

IV. Glass for Integrated Ion Exchange Devices

The glass constitutes the basic material for the fabrication of integrated ion exchanged devices. In order to control and develop this technology, it is necessary to know the structure and properties of glasses. The good selection of the glass composition, have an influence upon fabrication conditions and on the qualities of the realized components. If we want to define an ideal glass for ion exchange, the selection criterion can be summarized in this way:

- the transparency in the visible and near – infrared must be as higher as possible;
- the refractive index must be close to that of silicon optical fiber. In order to minimize the losses due to the non adaptation of refractive index (Fresnel reflexion) during the connection of those elements between them;
- the glass must be homogeneous, without bubbles, and unconstrained;
-the glass must resist chemically to the water and acids to make easier the manipulation and to improve the life duration of elaborated components;
- the glass must resist to the thermal shocks and the temperature of vitreous transition (Tg) must be sufficiently high to envisage the fabrication of optical devices at temperatures of 300 – 500 ^0C ;
- the glass must have an electrical conductivity at high

Table II. Parameters and characteristics of exchanged waveguide with different ions

Ions (molten salt)	*maximum refractive index*	*ion exchange parameters (time diffusion and*	*propagation losses dB/cm*	*remarks*

	change (Δn)	temperature)		
Li^+	0.02	seconds, >500 ^0C	>1	high tensile stress
K^+	0.009	minutes, ~400 ^0C	<0.2	compressive stress
Rb^+	0.01	hours, ~500 ^0C	>1	high price
Cs^+	0.04	hours, ~400 ^0C	~0.1	slow diffusion
Tl^+	0.1	minutes, ~500 ^0C	<0.1	additional expenditure for safety
Ag^+	0.1	seconds, ~350 ^0C	<0.2	low thermal stability

Table III. Characteristics and diffusion parameters of exchanged ions in glasses

Glass	Exchanged ions	Δn (*)	T (^0C)	D (**) μm^2/min	μ (***) μm^2/V..min	Losses dB/cm	Ref.
Soda-Lime	Ag^+/Na^+	0.09	375	0.42	-	/	[18]
			330	0.15	5.0	/	[19]
Corning 0211	=	0.049	343	0.12	/	<0.2	[20]
Alumino-boro-silicate	=	0.122	270	0.25	18026	<0.2	[21]
BK7	K^+/Na^+	0.009	385	0.085	/	0.07	[22]
Soda-Lime	=	0.008	=	0.065	1.63	/	[23]
Pyrex 7740	=	0.007	=	0.037	/	/	=
Corning 0211	=	0.005	400	0.14	/	<0.1	[24]
Fisher Premium	=	0.008	=	0.03	/	1	=
B270 schott	=	0.008	450	0.18	/	<0.2	[25]
			400	0.048	7		
KF3	Tl^+/Na^+	0.054	414 – 480	/	/	<0.1	[26]
BGG21	Cs^+/K^+	0.04	/	/	/	<0.1	[27]

(*) Δn: maximum of refractive index variation, (**) D: Diffusion coefficient, (***) μ: Electrical mobility.

temperature (200 – 500 ^0C).

The silicate glass satisfies the majority of the quoted criteria. It is well used for the fabrication of passive integrated waveguides. If it contains rare earth ions as erbium, neodymium, ytterbium, etc…it is also used for the fabrication of active integrated devices as lasers, amplifiers etc...

The alkaline ions as the sodium, potassium, cesium, lithium, rubidium and the monovalent ions as silver and thallium are the good candidates for the ion exchanges. The composition of glass has an important role on the exchange

kinetic, refractive index variation and the performances of obtained structures. For each type of exchanged ions are developed some specific glasses, especially for Ag+, K+, Cs+

and Tl+. In this part, we give a review on some of them.

A. Glass for silver-sodium ion exchange

The ionic radius of silver is close to that of sodium, which can make easier the mobility and the diffusion during the exchange at moderate temperatures (200 – 350 ^0C). The Tg of this kind of glass can be more than 500 ^0C. It contains an important quantity of sodium and exempt from impurities as Fe +++ or As ++. This is mainly because glasses contain impurities that react with Ag+ ions and compound the loss problem. The table 4 gives the compositions of some glasses used for silver ion exchanges.

The refractive index variation depends on silver nitrate dilution in the molten salt and the quantity of sodium in glass. The molten salt is composed from sodium nitrate and silver nitrate.

The saturation level of refractive index is obtained with 10 (mol.% .) of silver nitrate for WG10 glass.

The characteristics of glasses mentioned above with silver – sodium ion exchange are given in table 5.

The diffusion efficiency depends also on sodium oxide content in glass and diffusion temperature. The presence of Al2O5 in glass increases this parameter. Many glass properties change when a second alkali is added in glass.

Properties mostly affected are those associated with alkali ion movement such electrical conductivity and loss, alkali diffusion, internal friction, viscosity and chemical durability. The introduction of a second oxide former as B2O3 increases the chemical durability and decreases the dilatation coefficient. If the diffusion coefficient of silver in glass is too large, that can have an influence upon the lifetime of elaborated components at low temperature. Excellent results are obtained with WG10 in terms of propagation losses (0.13 dB/cm), possibility of burying the waveguide, mechanical and chemical resistance Fig. (3) [28] [29].

B. Glass for potassium-sodium ion exchange

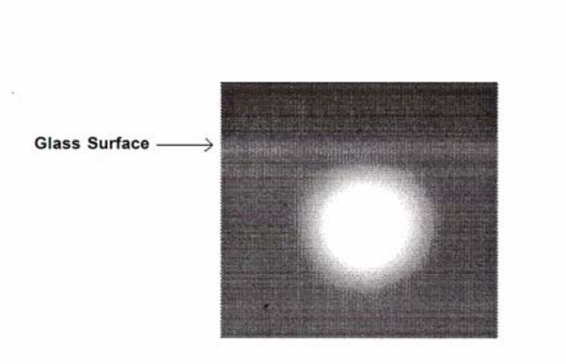
The potassium is stable and the obtained waveguides with this exchanged ion is reproducible. The temperature of exchange is larger than $400\,^{0}C$, due to the important size of potassium. The large difference between the potassium and sodium ions causes birefringence due to induced surface stress. This birefringence can be exploited to obtain components with maintain or separation of polarization. The Tg of glass is relatively higher. Some specified glass compositions are used for ion exchange (table 6).

Table IV. Glass compositions used for silver-sodium ion exchange

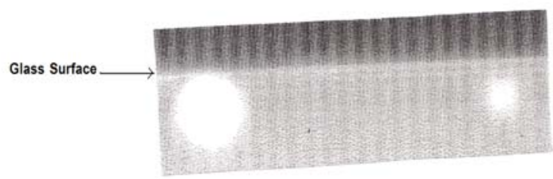
Glass composition	*Corning 0211 Wt.%*	*WG10 Wt.%*	*Alumino.boro.silic. Mol.%*	*LIES1 Mol.%*	*LIES2 Mol.%*	*LIES3 Mol.%*	*LIES4 Mol.%*
SiO_2	65	63	37.5	60	60	60	60
Al_2O_3	2	12	25	/	/	/	/
Na_2O	7	13	25	20	17	15	5
K_2O	7	/	/	/	3	5	15
Li_2O	/	0.5	/	/	/	/	/
MgO	/	2.5	/	/	/	/	/
CaO	/	4	/	/	/	/	/
TiO_2	3	/	/	/	/	/	/
B_2O_3	9	5	12.5	20	20	20	20
ZnO	7	/	/	/	/	/	/
Ref.	[14]	[22]	[15]	[22]	[22]	[22]	[22]

Table V Characteristics of silver- sodium exchange in glasses

Glass	*Dilution of Ag+ in molten salt (mol.%)*	*Δn max*	*T °C*	D_{Ag+} $\mu m^2/\min$	*Activation energy KJ/mol*	*Ref.*
Corning 0211	/	0.049	375	0.42	/	[14]
WG10	10	0.695	350	1.2	17.72	[22]
Alumino Boro. silicate	/	0.122	270	0.25	/	[15]
LIES1	0.2	0.046	350	3.25	66.1	[22]
LIES2	1	0.042	=	0.31	71.1	[22]
LIES3	1	0.31	=	0.18	72.9	[22]
LIES4	1	0.024	=	0.1	81.5	[22]



(a)



(b)

Fig.3 an integrated buried waveguide (a) integrated buried coupler (b) elaborated on WG10 glass

Table VI Compositions of glasses used for potassium-sodium exchange

Glass composition	*Corning 0211 Wt.%*	*BK7 Wt.%*	*PYREX 7740 Wt.%*	*B270 Schott Wt.%*
SiO_2	*65*	*69.6*	*81*	*73.5*
Al_2O_3	*2*	*/*	*2*	*2.3*
Na_2O	*7*	*8.4*	*4*	*2.6*
K_2O	*7*	*8.4*	*/*	*12.6*
BaO	*/*	*2.5*	*/*	*/*
MgO	*/*	*/*	*/*	*2.4*
CaO	*/*	*/*	*/*	*6.6*
TiO_2	*3*	*/*	*/*	*/*
B_2O_3	*9*	*9.9*	*13*	*/*
ZnO	*7*	*/*	*/*	*/*
traces		*1.5*	*/*	
Ref.	*[14]*	*[16]*	*[17]*	*[19]*

The diffusion time of potassium in glass is moderate (30 – 60 min) and it is difficult to bury the component under the surface glass. The surface guided structure is characterized by a supplementary propagation and coupling losses.

A. Glass for Cesium-potassium ion exchange

The cesium has an important ionic radius; it is preferable to use glass which contains high content of potassium. It is a stable ion but moves slowly. The exchange induces an elevation of refractive index about 0.04. The exchange temperature is appreciably high. The best results in term of propagation losses and bury waveguide are obtained with BGG 21 glass. The glass composition is unknown. The glass is composed of SiO2, Al2O5, B2O3 and K2O (12.5 mol.%.) [27]. It is produced specially for Cesium exchange. British glass developed a glass for cesium exchange called WG 16. It consisting of SiO2 (0.423), B2O3 (0.064), Al2O5 (0.261), Na2O (0.01), K2O (0.195), F (0.08), As2O3 (0.002) and Sb2O3 (< 0.0002). No result is published for this type of glass.

B. Glass for Thallium –sodium or potassium ion exchange

The thallium ion is characterized by a very high electronic polarisability in comparison with that of other alkaline ions. This induces an important refractive index elevation. The variation of refractive index can reach $\Delta n=0.1$. The thallium is chemically stable, the propagation losses is about 0.1 dB/cm. However, it is very toxic. The uses of thallium in ion exchange require special safety equipments. Its size imposes a diffusion temperature range of 400 to 550 ^0C. The waveguides obtained by thallium ion exchange have excellent optical performances. The thallium – potassium exchange offer an important variation of refractive index without constraints. It can result a constraint with thallium – sodium ion exchange. For this raison, the specific glass for thallium ion exchange must contain an important quantity of potassium oxide. Some glass is developed for thallium ion exchange as KF3 glass [26] and Alcatel glass for thallium exchange. The composition of the glass is unknown, but it contains SiO2, Al2O5, B2O3, K2O, Na2O, and F.

C. Criteria of glass selection.

After this review on glass for integrated optics, we can try to propose selection criteria of glasses which fulfill the conditions mentioned in the introduction.

The basic former is the silicon. It is characterized by its very well transparency in the visible and near infrared ranges. The silicon improves the behavior of the glass to chemical and mechanical shocks, but the elaboration temperature stays high (superior than 1700 ^0C) as well as the electrical resistivity and the viscosity.

The addition of alkaline oxides permits to reduce the elaboration temperature, the viscosity and the chemical durability of glass. Moreover, the presence of alkaline oxides increases the dilatation coefficient, the electrical conductivity and the refractive index. A small quantity of fluoride reduces notably the refractive index.

The introduction of B2O3 increases the chemical resistance and decreases the dilatation coefficient.

The CaO and BaO improve also the chemical resistance of glass.

The addition of Al2O3 for a ratio of Al/Alkaline less than 1 increase the work interval and the diffusion coefficient of alkaline ions, improve the chemical and mechanical resistance, and reduce the demixion tendency.

The PbO must be avoided; it increases the refractive index and the electrical resistivity, decreases the Tg and helps the separation of phases.

The B2O3 and Na2SO4 are introduced with low percentages in order to improve the fusion reactions and eliminate the released gazes.

Finally, the transition elements (Ti, V, Cr, Mn, Fe, Co, Ni, and Cu) must be avoided due to their absorption bands.

V. Conclusion

Integrated optical on glass has reached today a degree of maturity, which will open the door for many applications fields. The more important application fields which can actually benefit of the development of the glass integrated optics are:

- optical signal processing,
- optical sensors,
- optical memories,
- optical communications.

Some integrated optical components are commercialized as well as optical dividers, multiplexers, demultiplexer, filters, sensors for different applications (mechanical, chemical, medical, military, astronomy...), etc…

The basic substrate of these devices is of course the glass. Many industrial companies in this field, develop their proper glasses, as well as corning glass, schott-IOT, teem photonics, etc…The composition of these glasses stay exactly unknowns for the reason of confidentiality.

The technique of ion exchange is well known and well controlled. The problem resides in the properties of glass, which is not well controlled. The future development of this technology must take in consideration the composition of glass. The composition will be adapted to the diffused ion, permits the control of the fabrication parameters and the performances of the elaborated devices. If compatibility with modern glasses and safety of process are considered important factors, K+ and Ag+ ion exchanged glasses present the most feasible options. The glass reproducibility, thermal stability of ion exchanged component and internal losses can be the axes of the improvement.

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