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FLUORESCENT Er_2O_3 DOPED LEAD SILICATE GLASS FOR OPTICAL AMPLIFIERS

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A hot-pressing method is investigated for the fabrication of a planar optical waveguide amplifier. Therefore commercially available LaSFN15 produced by Schott is used as substrate and cladding material in combination with Er_2O_3 doped lead silicate glass as core material, synthesised by a hybrid sol-gel melting technique. The lead silicate glass is selected for its low melting temperature required for the waveguide processing. The core glass is adapted to the LaSFN15 with respect to the thermal expansion coefficient and the refractive index. The composition suited for the hot-pressing technology is evaluated to be 45 mole% PbO and 55 mole% SiO_2. The influence of increasing Er_2O_3 content to these parameters is compensated by changing the molar ratio of PbO/SiO_2. The optimal composition of the doped core glass is found out to be a glass containing around 3 wt.% Er_2O_3, because concentration quenching occurs above.

1. Introduction

Optical waveguides consisting of inorganic materials can be realised by several well known techniques like ion exchange, field assisted ion exchange MeV ion implantation, flame hydrolyses, chemical vapour deposition, laser deposition, sputtering and the recently published hot pressing technique [1]. Each processing technique demands special materials with adjusted properties to allow the fabrication of linear waveguides. Realising planar optical amplifiers leads to even more constraints of the optical glass required. In our work we decided to use the hot-pressing method for the waveguide preparation, which has potential for low cost devices and allows to confine doped material to the waveguide core. Doping only the waveguide core leads to reduced threshold of erbium doped amplifiers, because in uniform doped waveguides the weakly pumped trivalent erbium in the cladding is absorbing in the spectral range around 1550 nm. Another way to lower the threshold and to rise the pump light absorption is the incorporation of ytterbium for an efficient energy transfer to the erbium ions.

For the hot-pressing method it is necessary to develop two types of glass: One for the substrate and the cladding and a glass for the waveguide core of higher refractive index, which can be doped for the use in optical amplifiers. These glasses have to be matched in a sense, that the coefficients of expansion differ only slightly and the refractive index of the core glass is about 3 % higher than that of the cladding and the substrate. Further the hot-pressing procedure requires that the viscosity of the core glass is considerably lower at the pressing temperature, than that of the substrate and the cladding. Therefore low melting glass is needed, that strongly changes its viscosity with increasing temperature. For optical waveguide amplifiers besides the described material properties high erbium concentrations are of great interest to achieve short, efficient pump light absorbing- and high gain amplifiers. For short amplifiers concentrations of more than $5 \cdot 10^{20}$ Er^{3+}/cm^3 are aimed at and therefore to be investigated for concentration quenching effects.

With respect to these requirements we concentrated on lead silicate glass in combination with the commercially available LaSFN15 glass produced by Schott. For the preparation of the lead silicate glass a special sol - gel hybrid synthesis is applied [2]. This route allows to achieve homogeneous products, while decreasing the melting temperature by 200 K to 300 K, reducing the PbO loss and preventing the Er_2O_3 agglomeration. Concerning the optical properties ultrapure precursors and dry air melting inside a silica furnace were used to minimize absorption and non radiative loss by impurities like OH-groups or Fe ions in erbium doped lead silicate glass.

The aim of this work is to evaluate the technology for fabricating a planar optical waveguides with Er_2O_3-doped lead silicate glass as core material and the commercially available LaSFN15 as substrate and cladding material. To achieve this aim, it is necessary to dope the adapted lead silicate glass with Er_2O_3. In order to keep the thermal expansion coefficient and the refractive index in the required range for LaSFN15, the lead content is adjusted. The synthesised glass is further to be investigated to avoid phase separation during the waveguide fabrication .

2. Experimental and discussion

2.1 Lead silicate synthesis

In this work lead silicate glass is adapted to the high number of requirements of the hot-pressing method, with respect to optical amplification. Those requirements are the refractive index difference, the matching of the coefficients of expansion, Rare Earth doping as far as viscosity differences of the core and cladding glass. Therefore a lead silicate glass with 45 mole% PbO and different amounts of lanthanide oxide (Er_2O_3 / Yb_2O_3) is prepared by the hybrid synthesis route [2].

The lead silicate glass is synthesised starting from lead(II)acetate, tetraethyl orthosilicate and lanthanide acetate. The acetate precursors were dissolved in 10 % acetic acid and the tetraethyl orthosilicate was hydrolysed and condensated at 60 °C for 3 hours. The gel was dried and the residual organics were removed. The organic free xerogels were melted at temperatures from 650 °C to 1200 °C in pure alumina crucibles and poured into graphite moulds.

2.2 Adaptation of material properties.

According to the typical range of refractive indices, coefficients of expansion, length and melting temperatures [3] of undoped lead silicate glass, LaSFN15 is selected for the substrate and cladding material. Based on this data the appropriate amount of lead (II) oxide for the expansion matching and adjustment of the refractive index difference is determined.

Table 1 shows a comparison of several material properties of LaSFN15 and different compositions of lead silicate glass. More detailed data can be observed in Fig. 1, containing the dependency of the refractive index and coefficient of expansion to the lead oxide content. An increase of the lead oxide content leads to a reduction of the melting temperature, to a faster decrease of the viscosity with rising temperature, a rise in the refactive index caused by the high polarizeability of the lead(II) ion and an increase of the coefficient of expansion by weaker bindings.

Tab. 1: Properties of the different lead silicate glass

Er_2O_3 [wt. %]	Compound	Composition [mole %]	n_e	T_g [°C]	$\alpha_{30-300)}$ [10 6·K^{-1}]	Fluorescence lifetime [ms]
0	LaSFN15	- -	1.88	680	7.3	-
0	PbO SiO_2 Er_2O_3	45.00 55.00 0	1.89	445	7.8	-
0	PbO SiO_2 Er_2O_3	56.8 43.2 0	1.95	380	10.1	-
1	PbO SiO_2 Er_2O_3	45.53 54.12 0.35	1.90	449	7.77	11,0
2	PbO SiO_2 Er_2O_3	45.71 53.57 0.72	1.89	452	7.82	11,7
3	PbO SiO_2 Er_2O_3	45.91 53.00 1.09	1.89	455	7.65	12
4	PbO SiO_2 Er_2O_3	46.11 52.43 1.46	1.90	464	7.75	10,5
5	PbO SiO_2 Er_2O_3	46.30 51.80 1.90	1.91	469	7.88	9,5

The influence of the erbium content up to 5 wt.% ($7 \cdot 10^{20}$ Er^{3+}/cm^3), concerning the density and the thermal expansion coefficient α is not significant , but T_g increases by 20 K to 30 K (Tab. 1). The refractive index also changes with the erbium oxide content up to 1.97 for 5 wt.% Er_2O_3. For the erbium doped materials the influence of the erbium concentration on the refractive index is compensated by a change in the amount of PbO (Tab. 1).

Concerning the hot-pressing procedure, the temperature from which tensions occur is the glass transformation temperature, where the viscosity of the core glass is to low to give way the contraction of the cladding / substrate material.

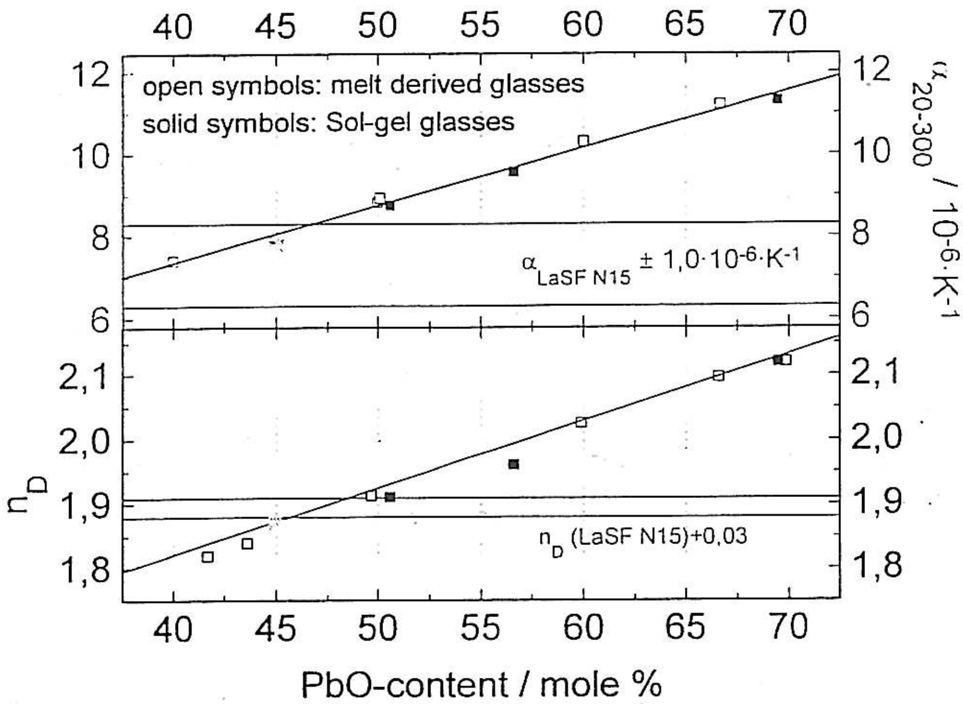


Fig. 1: Adaptation of lead silicate glass properties to LaSFN15

The resulting range of possible expansion coefficients suited for the LaSFN15 glass is marked in the upper part of the diagram. These values result from an estimation based on the work of Hogan et. al. [4] .

$$\Delta\alpha \cdot T_{g\,core} \leq 500 \cdot 10^{-6} \qquad (1)$$

Applying a core material with a T_g of less than 500 °C and using LaSFN15 as substrate, possible values for α result from $6{,}3{\cdot}10^{-6}\,K^{-1}$ to $8{,}3{\cdot}10^{-6}\,K^{-1}$. This restricts the use of lead (II) oxide contents to concentrations of less than 47 mole% for the application in waveguides made by the hot-pressing method.

2.3 Reduction of non radiative relaxation

To reach effective amplification in a glass it is necessary to obtain high $^4I_{13/2}$ - $^4I_{15/2}$ fluorescence lifetime of Er^{3+} ion. Therefore radiationless processes from the $^4I_{13/2}$ state like multiphonon relaxation with the glass host and quenching processes by impurities must be avoided. The use of ultrapure precursors and melting in dry air atmosphere in a silica tube lead to a reduction of the OH content to 15 ppm and the Fe content to around

20 ppm. By this measures lifetimes of more than eleven milliseconds were achieved for concentrations up to 5 wt% (Tab. 1). The decrease of the fluorescence lifetime for Er_2O_3 contents from 3 to 5 wt% can be contributed to concentration quenching. Resulting from the observed concentration quenching at around 3 wt%, concentrations below this value are to be used favourably for the fabrication of amplifying waveguides.

2.4 Waveguide fabrication

The first step of the waveguide fabrication process (Fig. 2) is the patterning of the substrate. This can be done by mechanical procedures like sawing, hot embossing or a physical process like ion beam etching. Substrate patterning by reactive ion etching is not suitable for LaSFN15, consisting of components, which form non volatile compounds. Starting with the basic evaluation of the hot-pressing process for the new combination of lead silicate glass, substrates patterned by sawing were used, because this was the only process fast available for LaSFN15.

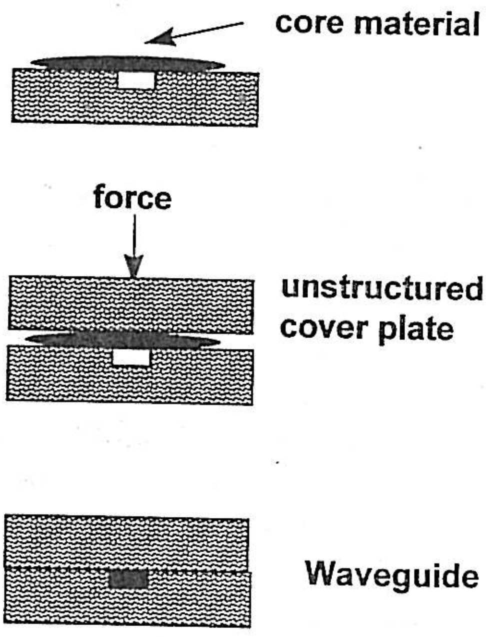
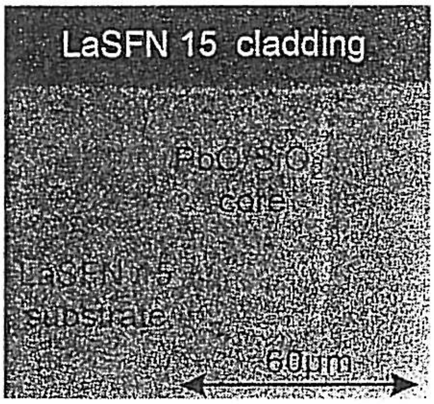


Fig. 2: The hot - pressing procedure

The waveguide is formed by pressing the core material into the structures at temperatures above the softening point, almost at T_g of the substrate. Therefore the sample consisting of substrate core glass and cladding, embedded between 3 mm thick carbon sheets, is softly pressed together for better heat conduction over the sample and heated to 680 °C. The heating rate of 25 °C/min is chosen in such a way that the heat distribution over the sample is nearly homogeneous. A plate of cladding glass is used as the pressing stamp to press the core material into the structures. The pressure was gradually increased to about 3000 N and applied for 20 min.

The Surplus of the core glass is thereby squeezed out sideward leaving only traces in the gap between substrate and cladding, which can not be seen looking at the crossection (Fig. 3). The substrate and cladding glass are bonded together at higher temperatures than the pressing is done, exerting low pressure. After bonding the substrate and cladding together the sample is rapidly cooled to T_g of the core glass. From this temperature fine cooling takes place.

A result achieved by this process is shown in figure 3. In the middle of figure 3 the crossection of the lead silicate core can be observed. The geometry results from the sawing procedure with a 30 µm dicing blade. A depth of 60 µm is achieved, with an sharp edge at the right side and a left sidewall of less quality caused by the blade. The rough sidewalls can be contributed to brittleness of LaSFN15, which is unfavourable for sawing. Looking at the top of the waveguide core, no remaining traces of the core glass can be observed, representing the properly evaluated hot-pressing parameters.



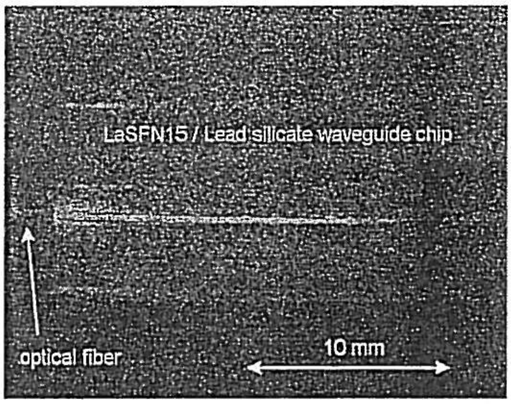


Fig. 3: a.) Polished endface of lead silicate LaSFN15 waveguide
b.) 980 nm pumped waveguide / ESA and scattered light

In figure 3 b. the 980 nm pumped waveguide chip is seen in the top view. The bright line represents the upconversion of the erbium ions which is caused high pump power and the scattered pump light. The inhomogeneities on that line are likely to be caused by inhomogeneities of the core glass, physical disturbances in the channel or at the waveguide sidewalls. Gain was not to be expected and measured in this large structures, because the inversion could not be reached.

3 Conclusions

A new set of glass suitable for the hot-pressing technology has been found. Adapted glass with different amounts of erbium oxide up to 5 wt% could be synthesised, meeting the requirements for the waveguide fabrication. These materials have been obtained by dry air melting in a high optical quality represented by the high fluorescence lifetime. On the bases of the thermomechanical properties the waveguide fabrication processing parameters have been obtained and fabrication technology successfully demonstrated. These results give the basis for further elaboration of investigated technique, by means of IBE or hot-pressed microstructured substrates containing structures in the needed sizes for amplifiers of around 4 µm by 4 µm.

4. Aknowledgement

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5. References

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